Internal Operating Procedure: Operations Directorate

Monitoring Branch

Title: DAAMS Tube Acceptance Testing

Division: Chemical Operations

Branch: Monitoring Branch

Building/Area: E3330 and other areas

This internal operating procedure covers operations, methods and procedures of a general nature not covered by a standing operating procedure. This procedure will be effective until rescinded or superseded.

Changes to this procedure will be accomplished by submission of revisions or amendments for approval.

Originator	Jill M. Meuser	Date
Analytical Team Leader	Steven D. Norman	Date
Monitoring Branch QA Coordinator	Paul Stewart	Date
Monitoring Branch Chief	Thomas E. Rosso	Date

Date: April 22, 2002

IOP Number: MT-4

Revision Number: 0

Prepared by: J. M. Meuser
X6731

Approved by: T. E. Rosso
X8428

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Signature Page

Title: DAAMS Tube Acceptance Testing

Operator's Statement: I have read, or have had read to me the procedures in this IOP. I, by my signature below, indicate that I thoroughly understand and agree to abide by these instructions.

Print Name	Signature	Date

Supervisor's Statement: I have personally reviewed this IOP and, to the best of my knowledge, believe that the information listed herein is correct.

Supervisor: _____ **Date:** _____
T. E. Rosso

DAAMS Tube Acceptance Testing

1.0 Purpose

To ensure that DAAMS tubes procured from vendors meet acceptance criteria as specified in the Monitoring Branch Quality Control Plan for Chemical Agent Standard Analytical Reference Material (CASARM), this procedure specifies the tests that must be performed.

2.0 Applicability

This IOP applies to DAAMS tubes purchased from vendors who do not provide acceptance testing or test documentation that meet CASARM requirements.

3.0 Receiving

When an order of tubes is received from a vendor, the tubes will be delivered to a Quality Assurance representative who will ensure that the order is complete. She/he will sort the tubes by manufacturer's lot number and complete a DAAMS Tube Acceptance Lot Specification Form (MBFORM-55), which lists each tube number in the lot. The packing slip and DAAMS Tube Acceptance Lot Specification Form (MBFORM-55) will be sent to the Chief of the Monitoring Branch.

After determining the number of tubes in the manufacturer's lot, the QA representative will determine the number of tubes that must be tested using the American National Standards Institute (ANSI)/American Society for Quality Control (ASQC) Z1.4, "Sampling Procedures and Tables for Inspection by Attributes" for an acceptable quality level (AQL) of 4%. The table of sample numbers and allowable defects is provided in the Chemical Agent Standard Analytical Reference Material (CASARM) Quality Assurance Plan for Chemical Agent Air Monitoring (CQAPCAAM).

The QA representative will randomly select the required number of tubes to be tested for each manufacturer's lot. She/he will enter the appropriate lot and tube number information on the DAAMS Tube Acceptance Testing Form (MBFORM-51). The DAAMS Tube Acceptance Testing Form (MBFORM-51) and the lot of tubes will be delivered to an analyst. The form will indicate the allowable number of testing failures at an AQL of 4%.

4.0 Testing

4.1 Visual Inspection

It is expected that one individual will perform visual inspection and pressure drop testing. This analyst must provide his/her name and the testing date in the appropriate section of the DAAMS Tube Acceptance Testing Form (MBFORM-51).

In accordance with the Monitoring Branch Quality Control Plan, each tube selected for testing will be visually inspected for loose packing, warped tube ends, or loose sorbent material outside the glass wool plug. The results of the inspection will be noted in the appropriate column of the DAAMS Tube Acceptance Testing Form (MBFORM-51).

If a sufficient number of tubes pass visual inspection, the tubes will be pressure drop tested, as described in Section 4.2.

If more than the allowable number of tubes fails the visual inspection, the lot is rejected. The analyst should return the entire lot to the QA representative. In consultation with the Chief of the Monitoring Branch, s/he will determine if each tube in the lot should be inspected. If so, new paperwork will be prepared and sent to the analyst.

4.2 Pressure Drop Testing

(This test is based on a procedure provided by PMCD, with modifications for Monitoring Branch sampling procedures.)

Pressure drop testing ensures that sufficient air can flow through the tube. Tubes will be tested using equipment comparable to that used during Monitoring Branch sampling activities. The flow rate used for testing will be the maximum rate used during Branch sampling activities, i.e., 500 ml/min.

A sampling pump with flow control is followed by a flow meter to assure a flow rate of 500 ml/min. The meter is followed by a vacuum gauge reading inches of mercury. The vacuum gauge is attached to the DAAMS tube and the pressure drop across the tube is read and recorded on the DAAMS Tube Acceptance Testing Form (MBFORM-51). For the 6-mm DAAMS tubes used at the Monitoring Branch, the acceptable pressure drop may not exceed 15 inches of mercury. For other tubes, criteria are specified in the Monitoring Branch QA Plan.

If a sufficient number of tubes pass pressure drop testing, the tubes will be conditioned and then tested for agent recovery, as described in Sections 4.3 and 4.4.

If more than the allowable number of tubes fail the pressure drop test, the lot is rejected. The analyst should return the entire lot to the QA representative. In consultation with the Chief of the Monitoring Branch, s/he will determine if each tube in the lot should be tested. If so, new paperwork will be prepared and sent to the analyst.

4.3 Tube Conditioning

After the representative tubes from a lot pass visual inspection and pressure drop testing, they will be conditioned in a flow of nitrogen at 290° C for 20 minutes, followed by a five minute cooling period. All tubes must be conditioned before agent recovery testing.

4.4 Agent Recovery Testing

Agent recovery testing ensures that the DAAMS sampling tubes can adsorb and desorb sufficient analyte for accurate sampling and analysis. The analyst performing recovery tests will enter his/her name and date on the appropriate line of the DAAMS Tube Acceptance Testing Form (MBFORM-51).

DAAMS tubes will be prepared according to the instructions in MB IOP-13 for GC/FPD QL samples. However, rather than a 3 µl spike, each tube will be spiked at one TWA for GB (4 µl spike of standard solution). The spiked tubes will be analyzed for GB according to IOP-13, including instrument calibration and data reporting. The results of agent recovery testing will be recorded on the DAAMS Tube Acceptance Testing Form (MBFORM-51). In accordance with CASARM requirements, recovery must be at least 75% to be acceptable. After the final test, the analyst/tester will sign the Acceptance Testing Form (MBFORM-51) to indicate whether the lot passes or fails the series of tests and deliver the form to the Chief of the Monitoring Branch.

If a sufficient number of tubes pass agent recovery testing, the remaining tubes in the lot will be conditioned and delivered to the Chief of the Monitoring Branch for issuance to samplers and analysts.

If more than the allowable number of tubes fail the agent recovery test, the lot is rejected. The analyst should return the entire lot to the QA representative. In consultation with the Chief of the Monitoring Branch, s/he will determine if each tube in the lot should be tested. If so, new paperwork will be prepared and sent to the analyst.

5.0 Recordkeeping

The Chief of the Monitoring Branch will maintain a notebook of purchasing, receipt, and testing data for DAAMS tubes. For each lot of tubes, the notebook will contain a copy of the purchase request, a copy of the packing slip, the completed DAAMS Tube Acceptance Lot Specification Form (MBFORM-55), and the completed DAAMS Tube Acceptance Testing Form (MBFORM-51). Any anomalies, observations, or decisions concerning lot acceptance will also be included.

July 2003

REVIEW AND APPROVAL OF QUALITY DOCUMENT

Title: DAAMS tubes monitoring procedures

IOP Number: IOP MT-11

Revision Number: 9

Date: October 2003

This form documents the review and approval of Monitoring Branch forms and quality documents, in accordance with the Monitoring Branch QC Plan and IOP MT-25.

This form will be effective until rescinded or superseded. Changes to this form will be accomplished by submission of revisions or amendments for approval.

Change Notice: Document format updated, perimeter pump arrangement revised. Sampling procedures amended and updated. Added appendix D.

Working copies of Revision 9 are available for review and/or printing from the MBFORMS directory on the Monitoring Branch Server.

Originator	Juanita Snyder	2 OCT 2003 Date
Team Leader	Juanita Snyder	2 OCT 2003 Date
QA Coordinator	Paul L. Stewart	2 Oct 2003 Date
Monitoring Branch Chief	Thomas E. Rosso	2 Oct 2003 Date

Distribution: Hard copy QAC for Forms book
Electronic master copy for MBFORMS directory on data file server
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Update the Master List on data file server
Scan new or updated document for release on data file server
Archive superceded copy (if applicable) on data file server

RECORD OF CHANGE

Previous revisions of this document did not incorporate a record of change page.

Revision 8 - IOP is being revised to include sampling procedure changes and equipment usage clarification. IOP is also being revised in a manner that will allow for one primary IOP concerning DAAMS monitoring procedures. Individual appendices shall contain the specific requirements that pertain to a particular site. Requirements can include site layouts, special equipment needs as well as flow rates, time interval and sample types specific to that site.

Revision 9 – Document format updated. Perimeter pump arrangement revised to allow for better overall sample coverage. Sampling procedures amended and updated. Addition of Amendment D (Pine Bluff Monitoring Procedures).

*Field Tags/Labels are used interchangeably in this document

Title: DAAMS Tubes Monitoring Procedures

Operator's Statement: I have read, or have had read to me the procedures in this IOP. I, by my signature below, indicate that I thoroughly understand and agree to abide by these instructions.

Signature	Date	Signature	Date

Supervisor's Statement: I have personally reviewed this IOP and, to the best of my knowledge, believe that the information listed herein is correct.

Supervisor: Tomas E. Rosso
T. E. Rosso

Date: 20 Oct 2003

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1.0 INTRODUCTION

This document presents the air sampling equipment and current procedures used by the Monitoring Branch, Sample Collection Team to collect chemical warfare materiel (CWM) air samples at Edgewood, MD and other locations.

1.1 PURPOSE

The purpose of this plan is to establish monitoring procedures that shall be used by the Edgewood Chemical Biological Center, Monitoring Branch sample technicians to set up sampling equipment and monitor the airborne concentrations of chemical warfare materiel (CWM) during the various type of monitoring procedures. Appendices will Contain sampling information pertaining to specific sites. All of the procedures contained within IOP MT-11 will apply to each appendix, but are modified to meet the specific requirements of the particular site. Modifications cover layout of monitoring locations, flows, time intervals and agents of interest to the specific site.

1.2 SCOPE

To establish the procedures and responsibilities for the sample technicians setting up and conducting air monitoring operations both at Edgewood, Maryland and at off-site locations. This plan describes the rationale for monitoring strategies for historical and confirmation air monitoring and choice of monitoring equipment.

*Field Tags/Labels are used interchangeably in this document

2.0 Sample Equipment

This section describes the procedures for setting up the sampling equipment prior to taking the actual sample. See section 4.0 for the procedures to follow at the end of the sampling period.

2.1A BIOS 6000 and Metalite pumps.

1. Set timers to required sampling interval (see Figure-3 for Times and flow rates).
2. Set delay time if require. If pump is not on electric power – delay time is limited to 10 minutes.
3. Attach dual sample port to tubing and then to the sample pump.
4. Remove DAAMS tubes form protective carriers. PRIOR TO USE – inspect tubes for flaws (i.e. chips, cracks or dirt in the frits). Do not use tubes if any flaws are found. Verify tube numbers are legible before using.
5. Insert the GLASS WOOL side of the DAAMS tube (see Figure-1) into the dual port sampler. The lowest number tube is placed into the port with the rubber band on it. If the rubber band breaks or is missing – replace it with a new one. Dual DAAMS tubes will be used concurrently during the monitoring procedure. The analysis of the DAAMS tube during desorption is a one-shot occurrence. If for some inexplicable reason the desorbed sample is not analyzed as anticipated, it cannot be recovered. Therefore, a second tube is used as a backup.
6. Adjust flow according to the sampling interval with a calibrated calibrator. Verify the calibrator has not expired. Flows must be within the specified range indicated in Figure-3. Before recording the three beginning flow rates, they must be within 10% of each other. Write the three flows on the field tag (see Figure-3).
7. Clear pump timer. If pump is not being used immediately, turn off the pump and place it on charging station.
8. If VX is being sampled – a VGPAD fitting is to be placed on end of each sample tube.
9. Pumps being used to sample for Lewisite are to be setup as follows:
    a. **DON GLOVES.**
    b. Set up according to steps 2.1.
    c. Once the pump is setup, turn on the pump and spike both tubes with 10uls of BME. Tubes may be spiked the night before, but not more that 24 hours in advance.
    d. Clear pump timer and remove gloves.
10. Pumps placement should be as close to the sample or area as possible without interfering with the operation or operators.
11. Secure equipment so it cannot inadvertently be knocked over.

2.1B Surveyor Pumps

1. Set timers to required sampling interval (see Figure-3 for Times and flow rates).
2. Set delay time if required. If pump is not on electric power – delay time is limited to 10 minutes.
3. Remove DAAMS tubes form protective carriers. PRIOR TO USE – inspect tubes for flaws (i.e. chips, cracks or dirt in the frits). Do not use tubes if any flaws are found. Verify tube numbers are legible before using.
4. Insert the GLASS WOOL side of the lowest numbered DAAMS tube (see Figure-1) into sample port numbered 1. Place the second tube (higher number) into the port identified as 2. Dual DAAMS tubes will be used concurrently during the monitoring procedure. The analysis of the DAAMS tube during desorption is a one-shot

*Field Tags/Labels are used interchangeably in this document

occurrence. If for some inexplicable reason the desorbed sample is not analyzed as anticipated, it cannot be recovered. Therefore, a second tube is used as a backup.

5. Adjust flow according to the sampling interval with a calibrated calibrator. Verify the calibrator has not expired. Set screw identified as "1" is used to adjust flow for sample port 1 with set screw "2" used to set the second tube flow. Flows must be within the specified range indicated in Figure-3 Before recording the three beginning flow rates, they must be within 10% of each other. Write the three flows on the field tag (see Figure-2).
6. Clear pump timer. If pump is not being used immediately, turn off the pump and place it on charging station.
7. If VX is being sampled – a VGPAD is to be used and placed on end of each sample tube.
8. Pumps being used to sample for Lewisite are to be setup as follows:
    a. **DON GLOVES.**
    b. Set up according to steps 2.2.
    c. Once the pump is setup, turn on the pump and spike both tubes with 10uls of BME. Tubes may be spiked the night before, but not more that 24 hours in advance.
    d. Clear pump timer and remove gloves.
9. Pumps placement should be as close to the sample or area as possible without interfering with the operation or operators.
10. Secure equipment so it cannot inadvertently be knocked over.

2.2 Sampling Harness Setup

Sampling harness is used with an electrically powered vacuum pump when monitoring multiple Labwaste, XXX or TAPC items that are located within engineering controls. Filter systems that use DAAMS as confirmation for MINICAMS alarms can also be configured with a sampling harness. A maximum of ten sample ports may be attached to form the harness, but can be adjusted for fewer samples. Client requests for XXX/TAPC and LABW will be setup, completed and samples turned into the analysis lab prior to starting another request.

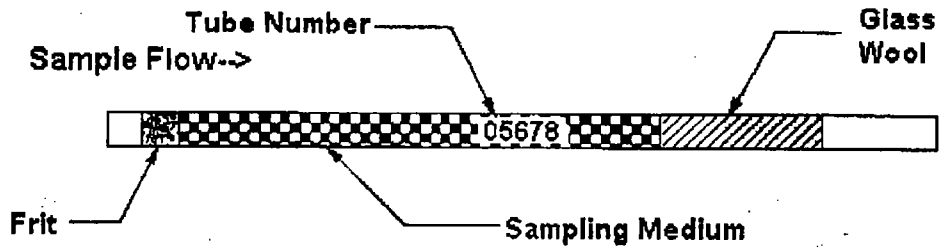
1. DON GLOVES.
2. Identify items. Barcodes affixed to items will be used as the sample identification (use last 6 digits of barcode). For off-site locations, the client's ID number will be used. If no barcode/ID is present, the ST will assign an item identification number in the following format (ymmdd###; example 20201001). The format indicates the year, month, day and the numerical sequence of the item. Item identification will be written on the item in permanent legible marker.
3. Place one set of DAAMS tubes by each sample.
4. Write the building number, item ID and the agent(s) being monitored on the field tag attached to the DAAMS tube carriers.
5. Make hole in the double bags of the item large enough for the DAAMS tubes to be inserted into and insert the DAAMS tubes. Verify numbers on tubes being inserted match those on the field tag. Repeat until all items have been identified on field tags and DAAMS tubes have been inserted into the bags.
6. Turn on the vacuum pump. The shortest allowable sampling interval shall be used for XXX/TAPC and LABW items. DO NOT MIX flow rates.
7. Start with the first item; adjust the flow to the appropriate flow rate for the proper time interval.

*Field Tags/Labels are used interchangeably in this document

8. Write the flow rates on the field tag and proceed to the next item. After all flows have been adjusted, go back and check flow rates on the first 3 items to verify they did not fluctuate out of the acceptable limitations.
9. If VX is being sampled – a VGPAD is to be used is to be placed on end of each sample tube after beginning flows have been recorded.
10. Ten minutes prior to the end of the sample period – remove the DAAMS tubes from the first item and record the ending flows (DO NOT ADJUST flows). VGPADs must be removed in order to obtain ending flow rates. Return tubes (replace VGPADs if required) into the first item and go to the next item and repeat the process until all flows are recorded.
11. At the end of the sampling period - turn off the vacuum source and pull the DAAMS tubes inserted into the first item out. Seal the hole in the bag with tape. Verify the DAAMS tubes pulled from the sample match the tube numbers written on the field tag.
12. Verify the sample ID written on field tag and the ID on item match. Remove DAAMS tubes from the dual port sampler and place them in the carriers, frit side down. Proceed to next item until all samples are collected.

2.3 Depot Area Agent Monitoring System (DAAMS) tubes

The components of the DAAMS tubes are identified in Figure-1. The DAAMS tube is always situated to draw the sample through the frit and onto sampling medium.



DAAMS Tube Components

Figure-1

2.4 Field Tags*

Field tags are hand written tags that contain information about the sample being collected.

1. DAAMS tubes numbers are written on the upper right hand corner of the field tag. **NOTE: Dual tubes will be used for all samples**. The lower numbered tube is listed first, with the second tube being listed below the first (see Figure-2 below). Visually verify written tube numbers match actual tubes.
2. Completed Field tags will contain the following information:
   1. Dual DAAMS tube numbers written on the upper right hand corner of the field tag. The lower numbered tube is always listed first, with the second tube being listed below the first (see Figure-2). Visually verify written tube numbers match actual tubes

*Field Tags/Labels are used interchangeably in this document

2. Three beginning flow rates written on the field tag (flows must be within the specified range indicated in Figure-3 and with 10% of each other) prior to collection of a sample.
3. Three ending flow rates written on the field tag for each tube at the conclusion of the sampling period. DO NOT make adjustment to ending flowrates. If flows cannot be obtained, an explanation is needed (i.e. broken tube). Identify large changes in flowrates from beginning flows.
4. Location where sample was obtained (i.e. building or site location).
5. Sample identification number; position placement or room number. QPs shall include a "Q" at the end of the sample identification (i.e sample is 30412001; QP would be 30412001Q; sample is POS.A; QP would be POS.AQ).
6. Time the sample started and ended. If the sample did not run the complete time interval, time should reflect actual time run.
7. Pump serial number should be written on lower right-hand corner of field tag.

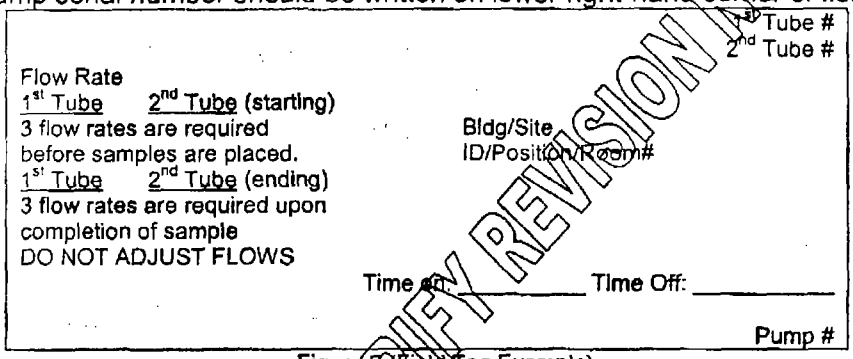


Figure 2 (Field Tag Example)

3.0 Sample Types, Times and Flow Rates

This section identifies various sample types, the allowable time intervals, corresponding flow rates and limitations (See Figure-3). Detailed information for individual sample types is located in the designated paragraph this section.

Sample Type	Description Dual tubes shall be used for ALL samples	Time	Target Flow	Flow Range Limits
1X Items (small)	Size should be limited to less than 3x3x3	48 min 1 hour	500mls/min 400 mls/min	480 – 520 mls/min 380 – 420 mls/min
1X items (large)	Larger than 3x3x3, 55 gal. Drums or items with complex internal parts Items should be completely wrapped. Extremely large items should have 2 samples taken at opposite ends	1 hour	400 mls/min	380 – 420 mls/min
Soil Samples	Sample jar of soil shall be double-bagged, with both bags sealed.	48 min 1 hour	500mls/min 400 mls/min	480 – 520 mls/min 380 – 420 mls/min
TAP Clothing	Items shall be double-bagged, with both bags sealed.	48 min 1 hours	500mls/min 400 mls/min	480 – 520 mls/min 380 – 420 mls/min
Monitoring identified in this section cannot be resampled. NOTE: Lower target flows do not have a lower limit.				
SOP	Sample pumps shall be placed as	4 hours	100 mls/min	100 – 110 mls/min

*Field Tags/Labels are used interchangeably in this document

Monitoring	close to the operation as possible without placing any equipment inside the hood.			Do Not Exceed lower flow limit
Background Monitoring (Historical)	A minimum of 2 positions shall be placed within a room. Hoods will require 1 pump.	2hours 4 hours 8 hours	200 mls/min 100 mls/min 50 mls/min	190 – 210 mls/min 100 – 110 mls/min 50 – 55 mls/min
Perimeter Monitoring	A minimum of 4 positions shall be set up (Pos.B, C, D and E) 5 hour time interval for sites working 10 hours days	2 hours 4 hours 5 hours 8 hours	200 mls/min 100 mls/min 80 mls/min 50 mls/min	190 – 210 mls/min 100 – 110 mls/min 80 – 88 mls/min 50 – 55 mls/min
Confirmation Monitoring	DAAMS tubes positioned next to the MINICAMS HSL (e.g. POS.A) or on filter bank	NTE 12 hours	500 mls/min	480 – 520 mls/min
3X Facility	A minimum of 2 positions placed in each room/hallway/egress area of the facility. Facility shall be monitored for a total of 24 hours	4 hours 8 hours	100 mls/min 50 mls/min	100 – 110 mls/min 50 – 55 mls/min
QP	A minimum of 2 per day. A set of QP DAAMS will run concurrently with designated samples. Times and flows will correspond with actual sample types identified in this table. All flow limitations noted shall also apply to QPs taken.			

Figure-3

3.1 General Area Air Monitoring

1. General area air sampling will be used for both first entry and quarterly period sampling requirements. Monitoring Branch personnel shall pay special attention to calibration, filling out of forms and chain of custody documentation.
2. Allowable times, flows and limitations are noted in Figure-4. DO NOT EXCEED noted flow or time limitations.
3. **NOTE: There are no lower limits for flow rates 100 mls or below.**

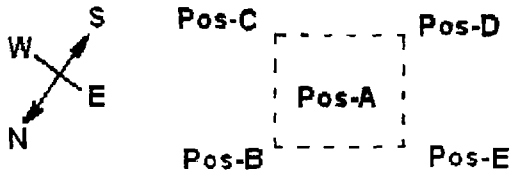
Sample Type	Description Dual tubes shall be used for ALL samples	Time	Target Flow	Flow Limits
General Area Air Monitoring (Historical)	1st Entry/Quarterly Storage and IHF	2 hours 4 hours	200 mls/min 100 mls/min	190 – 210 mls/min 100 – 110 mls/min

Figure-4

3.2 Background/Perimeter Monitoring

1. Outdoors - the Sample Technician (ST) shall assess the area and set up a minimum of three positions, more may be set up if deemed necessary in the judgment of the ST. Positions should be approximately 12 – 30 feet apart and set up approximately 50 feet from the source.
2. Perimeter monitoring shall consist of 5 positions. This will allow for fluctuating winds, without having to reposition sample pump positions due to wind shifts. The four outside positions shall be situated base on compass points (see Figure-5). Positions should be approximately 12 – 30 feet apart and set up approximately 50 feet from the source. Position "B" shall be place on the northern point of the perimeter with the other positions being place in a clockwise pattern. The fifth position (POS-A) shall be located within the exclusion area. The "A" position will be set using the Confirmation flow setting indicated in Figure-3 only if it is used to confirm readings obtained from MINICAMS heated sample lines. If the MINICAMS HSL is not used, the flowrates will coincide with the rest of the perimeter flow rates.

*Field Tags/Labels are used interchangeably in this document



Perimeter positions shall be place in a clockwise pattern around the exclusion area.

Figure-5

3. Indoors – the ST shall assess the area and set up a minimum of two positions, more may be set if deemed necessary in the judgment of the ST. For extremely small areas (10' x 10' or less), set one position using dual tubes.
4. Allowable times, flows and limitations must NOT EXCEED noted flow or time limitations. See Figure-6.
5. **NOTE: There are no lower limits for flow rates 100 mls or below.**

Sample Type	Description Dual tubes shall be used for ALL samples	Time	Target Flow	Flow Limits
Background or Perimeter Monitoring	Hoods require 1 pump and harness setup with no activity inside hood. Perimeter monitoring requires a minimum of 4 positions	2 hours 4 hours 8 hours	200 mls/min 100 mls/min 050 mls/min	190 – 210 mls/min 100 – 110 mls/min 050 – 055 mls/min
Perimeter Monitoring of Exclusion area	Exclusion area pump (Pos-A) is set with HSL of MINICAMS for confirmation of MINICAMS alarm	NTE 12 hours	500 mls/min	480 – 520 mls/min

Figure-6

3.3 Standard Operating Procedure (SOP) Monitoring

1. SOP monitoring is performed to verify the engineering controls during agent operations. Sample equipment will be placed as near the operation as possible without interfering with the operator or the operation. DAAMS tubes or equipment is not placed in the hood where agent work is being performed. Sampling port is fixed to the outside of the hood sash near the breathing zone of the operators conducting the operation.
2. Sample technician is to annotate the Laboratory Monitoring Record maintained in the laboratory room where the SOP is located.
3. Allowable times, flows and limitations must NOT EXCEED noted flow or time limitations. See Figure-7.
4. **NOTE: There are no lower limits for flow rates 100 mls or below.**

Sample Type	Description Dual tubes shall be used for ALL samples	Time	Target Flow	Flow Limits
SOP monitoring	Laboratory agent operations	4 hours	100 mls/min	100 – 110 mls/min

Figure-7

3.4 Labwaste/XXX TAPC items

1. Labwaste is any trash generated during laboratory agent operations that requires monitoring before disposal.

*Field Tags/Labels are used interchangeably in this document

2. XXX items are equipment or tools that have been used or potentially exposed to agent during agent operations.
3. TAPC is clothing or items that will be worn again. This includes reusable boots, gloves, coveralls, masks, respirators, uniforms, SCBA and associated equipment/harnesses. It does not include disposable coveralls, booties or gloves.
4. All of the above items must be double-bagged, dry and sealed for a minimum of 4 hours at an ambient temperature of 70 degrees Fahrenheit.
5. **Gloves will be worn** when sampling any of the items listed. Dispose of appropriately at conclusion of sampling procedure.
6. Client requests for XXX/TAPC and LABW will be setup, completed and samples turned into the analysis lab prior to starting another request.
7. Refer to section 2.0 for equipment setup procedures.
8. Barcodes affixed to items will be used as the sample identification. Serial numbers for client equipment can be use if requested. The ST will assign an item identification number in the following format (ymmdd####, example 20201001) if no barcode or specific client request is available. The format indicates the year, month, day and the numerical sequence of the item. For off-site items the number will contain 2 identifying characters from the site. Examples: Lowery site = LW20201001; Cleveland site = CL20201001.
9. Allowable times, flows and limitations must NOT EXCEED noted flow or time limitations. See Figure-8.

Sample Type	Description Dual tubes shall be used for ALL samples	Time	Target Flow	Flow Limits
Labwaste/XXX or TAPC items	PPE or trash generated during agent operations	48 min 1 hour	500 mls/min 400 mls/min	480 – 520 mls/min 380 – 420 mls/min

Figure-8

3.5 QPs

QPs are a set of dual DAAMS tubes spiked with known concentration of agent standard run for specific times and flow rates. Tubes are spiked with approximately 1 TWA of standard, not more that 30 minutes prior to starting actual sample. QPs run concurrently with actual samples for the exact times and flows that the samples are taken (i.e. SOP operation runs 4 hours @ 100 mls/min – QP will be set up identically and run concurrently). **QP field tags will be annotated with the agent(s) of interest and highlighted for easy recognition**. A set of QP tubes will be run for every 10-labwaste/XXX or TAPC items sampled (i.e. 22 labwaste items would generate 3 QP sets). Laboratory personnel will spike the appropriate number of QPs based on sample technician's request. QPs shall be logged in with associated samples as a batch. Two copies of the Scratchlog shall be printed. One copy of the Scratchlog (marked LAB COPY) will be placed with the samples associated with the QP in a Ziploc bag

3.6 Hot Box Procedures for Headspacing Items

The procedures for monitoring bagged or boxed items when weather conditions don't allow items to reach the minimum 70° ± 10 degrees Fahrenheit ambient temperature requirement. This procedure is used for Labwaste, XXX and TAPC items generated during off-site field operations. Sample equipment is set up following the procedures of Section 2.1 or 2.3.

1. Items shall be wrapped/double-bagged prior to acceptance and placing inside Hotbox.

*Field Tags/Labels are used interchangeably in this document

2. Barcodes affixed to items will be used as the sample identification. Serial numbers for client equipment can be used if requested. The ST will assign an item identification number in the following format (ymmdd###; example 20201001) if no barcode or specific client request is available. The format indicates the year, month, day and the numerical sequence of the item. For off-site items the number will contain 2 identifying characters from the site. Examples: Lowery site = LW20201001; Cleveland site = CL20201001
3. **DON MASK and gloves**
4. Place items in heated Hotbox. If items are drums/jars, the lids must be taken off in order for the contents to off-gas into the bags. Make sure not to place items near heat source.
5. Items are to be heated to 90 degree Fahrenheit, plus or minus 10 degrees for 15 minutes
6. Items are then to off-gas for an additional 15 minutes before starting the sampling process.
7. Sample equipment should be set according to procedures in section 2.0, insert DAAMS tubes through all layers of wrapping/bags. Use the appropriate time intervals and flow rates for the sample type indicated in section 3.0 and begin sampling.
8. Allowable times, flows and limitations must NOT EXCEED noted flow or time limitations. See Figure-9.

Sample Type	Description Dual tubes shall be used for ALL samples	Time	Target Flow	Flow Limits
Labwaste/XXX or TAPC items	PPE or trash generated during agent operations	48 min 1 hour	500 mls/min 400 mls/min	480 – 520 mls/min 380 – 420 mls/min

Figure - 9

9. At the conclusion of the sample period, remove DAAMS tubes from item and seal hole.
10. Use procedures in section 4.0 at the conclusion of the sampling period. Do Not make adjustments to ending flow rates. If flows cannot be obtained, an explanation is needed (I.e. broken tube). Identify extreme changes in ending flow rates from beginning flows. Proceed until all items are completed.
11. Remove Mask and gloves. Dispose of appropriately.

3.7 Soil Headspace Analysis Procedures.

Under normal conditions soil sample headspace analysis will be conducted using the MINICAMS. If DAAMS tubes are used in place of MINICAMS, start the following sequence at step 12. DAAMS tubes must be used to confirm all positive MINICAMS results above 0.2 AEL. This monitoring shall be used to screen soil samples for H and L contamination. Headspace monitoring for CG, CK or chloropicrin is not necessary due to the extreme volatility of these compounds. Headspace Monitoring Procedures of soil samples using the MINICAMS and DAAMS are as follows:

1. Contractor delivers soil sample to monitoring personnel with proper chain of custody documentation.
2. **Don protective gloves.** Don protective mask when soil sample is collected by personnel wearing EPA/OSHA Level C PPE or above.
3. Place up to six samples in a heated sample box. Open bags and remove sample jar

*Field Tags/Labels are used interchangeably in this document

lids. Close sample box lid and allow samples to equilibrate at 90° ± 10 degrees Fahrenheit for 15 minutes.

4. Insert temperature probe into a selected soil sample. Allow samples to off-gas an additional 15 minutes.
5. Using the procedures outlined in section 2.0 and flow rates and times interval indicated in Figure-10 collect soil sample. Make sure that frits of DAAMS tubes are not inserted into the soil sample.

Sample Type	Description Dual tubes shall be used for ALL samples	Time	Target Flow	Flow Limits
Soil Samples	Soil, stone, concrete sample generated from a CWM location	48 min 1 hour	500 mls/min 400 mls/min	480 – 520 mls/min 380 – 420 mls/min

Figure-10

8. Transport DAAMS tubes to the MAP for analysis by Dynatherm/GC/MS.
   a. If agent is detected from the DAAMS tubes analysis, the USAESCH On-Site Safety Specialist must be immediately notified.
   b. If DAAMS analysis results in a non-detect for agent, give clear samples to the contractor for proper disposition.
9. Soil Headspace Data Notification: The USAESCH On-Site Safety Specialist will be notified of all confirmed detection's. This includes concentration levels below the AEL value.
10. Soil Resample Procedure: In the event that soil has been decontaminated, the soil cannot be re-sampled using the headspace analysis procedure. Therefore, the ECBC shall request a sample of the decontaminated soil for extraction and subsequent analysis for H and L by GC/MS to confirm complete decontamination.

11. Soil Headspace Sampling Records. Copies of all soil headspace air sampling results will be maintained.

4.0 Completed DAAMS Tube Sample Collection

4.1 Before disconnecting the DAAMS tubes samples from the Gemini or harness:

1. DON GLOVES prior to collection of samples for all XXX, TAPC and labwaste items.
2. Verify DAAMS tube numbers are written on upper right hand corner of the field tag.
3. Verify the DAAMS tube numbers match the numbers written on the field tag.
6. Verify that pertinent sample information for each sample is written on the appropriate field tag (i.e. sample ID, ON & OFF times, beginning & ending flow rates, bldg number, agent(s) being sampled).
7. Verify actual sample is identified (use permanent marker and make legible).
8. Ensure ending flows were taken and recorded on the field tag. Recorded ending flows should be within 10% of each other. Do not make adjustments to the ending flows. Make a note of any notable increase or decrease in the flow rate on the field tag.
9. Return the DAAMS tubes (FRIT end first) to the protective carrier, cap carrier and place with the appropriate field tag.
10. Place completed sample(s) in a Ziploc bag along with the associate QP sample(s).
11. A government vehicle is used to transport finished samples to a Monitoring Branch laboratory.

*Field Tags/Labels are used interchangeably in this document

12. Sign into the Sample database and enter the sample information into the TAG/Horizon program and record flow information for each sample on the Air Monitoring Worksheets. **QP flow rates are to be recorded on a separate Air Monitoring Worksheet but are logged in with the samples they were collected with.** Verify all information was transposed correctly from the field tag to the flow sheet. Verify that sample identification assigned by computer matches on the Scratchlog, TAGS and flowsheets. The sample database programs generate all necessary forms and tags for samples. DO NOT use any other forms.
13. Completed samples should be logged in and turned over to the lab within **1 ½ hours of completion**. Exceptions will be made for sample obtained from remote locations.
14. Take samples to the laboratory/MAP, analysts will verify receipt and annotate the Scratchlog. Make sure all signatures, dates and times are written in blue or black ink at the bottom of the form. If no one is available to accept samples and sign the Chain of Custody on the Scratchlog – DO NOT LEAVE THEM. In the event the samples cannot be turned over to the laboratory/MAP, make a note on the flow sheets and store the samples in the refrigerator. The analyst will record any discrepancies or anomalies. These comments shall be added to the comment section of the database program.

5.0 Sample Documents

1. All pertinent data shall be recorded on the Scratchlog and collected in Bldg E3344. All data from off-site operations will be collected on a TAG computer at the site. Upon the return of computer the sample information will be downloaded and added to the data in the main computer.

2. The following required information is recorded in the appropriate location on the Scratchlog, personnel roster or the Air Monitoring Worksheet.

   a. Scratchlog (MBFORM-12)
      (1) Sample number (assigned by computer)
      (2) Building or off-site location
      (3) Sample identification number (Item, Position, Room or Barcode)
      (4) Date of sample
      (5) Agent(s) being tested for
      (6) Type of sample (background, SOP, TAPC etc.)
      (7) Time ON and OFF for samples
      (8) Point of contact and phone number (requestor's last name)
      (9) Signatures of two STs and lab personnel, along with date and time samples were received.

   b. Air Sample Workorder (MBFORM-39)
      (1) Sample number
      (2) Building or location
      (3) Sample identification (Item/position/room)
      (4) DAAMS tubes numbers (lowest number listed first)
      (5) Three Flow rates for all tubes prior to sample start
      (6) Three Flow rates for all tubes at conclusion of sample run
      (7) Start and End times for samples

*Field Tags/Labels are used interchangeably in this document

(8) Sample type (i.e. LABW; SOP) SOP monitoring requires the SOP number(s) be recorded on the form.
(9) Comments on any discrepancies concerning tube flows (i.e. tube broken, wet, dirty)
(10) Signature and date of two ST annotated on the form.
(11) Client's last name and organization. Client and organization for QP samples is always Rosso and QC Account

c. Personnel Roster (MBFORM-42) (For laboratory operations at ECBC)
(1) Building and room number
(2) Date of Monitoring
(3) Agent(s) being tested for
(4) SOP number(s)
(5) Point of contact and phone number (last name only)
(6) Time sample pump started and ended
(7) Name, signature and full social security number of personnel present during time of monitoring.

3. Two QPs tubes will be collected for every ten samples collected during sampling process to ensure that no tampering or sampling vagaries occur (example: if 20 samples are collected, a total of 4 QP tubes should be generated). A QP is a sample spike with a known concentration of analyte and tracked through the analysis. The results are used to gage the performance of the system.

4. Sample labels are generated from a PC database program. The sample labels are attached to the field tag and assigned set of DAAMS tubes. The label is removed by the analyst and affixed to the corresponding chromatogram.

5. When the Foxpro sampling program is used, the sample technician is responsible for printing all documents (Scratchlogs, Sample Tags and Datasheets). The analyst will annotate the datasheet with the date the samples were analyzed, the sample results and a signature. The analyst will enter analyte results into the monitoring database. The sample technician is responsible for ensuring the information is on the datasheet. Return it to the analyst if incomplete.

6.0 Notification

Notify both the Safety Division and the Health and Veterinary Services Office of any result above the 8-hour time weighted average (TWA) airborne exposure limit.

7.0 Air Sampling Records
Maintain all sampling records per AR 40-5 requirements.

8.0 Transport

Transport of completed DAAMS samples will be accomplished in a government vehicle.

*Field Tags/Labels are used interchangeably in this document

MT-11, Appendix A: Lauderick Creek

All equipment setup and monitoring for the Lauderick Creek site will be in accordance with procedures indicated in MT-11. Specific flow rates and time intervals for Lauderick Creek sampling procedures are indicated in figure-1 of this appendix.

Monitoring Flow and Sample Rates Dual DAAMS tubes shall be used for all monitoring				
Sample Type	**Description and preparation procedures prior to sampling**	**Sample Time**	**Target Flow Rate**	**Acceptable Flow Limits**
1x Items (small)	Item should be double-bagged and less than 3x3x3 feet	48 min 1 hour	500 mls/min 400 mls/min	480-520 mls/min 380-420 mls/min
TAPC	Items shall be double-bagged and sealed	48 min 1 hour	500 mls/min 400 mls/min	480-520 mls/min 380-420 mls/min
Soil Samples	Item shall be double-bagged and identified prior to sampling	48 min 1 hour	500 mls/min 400 mls/min	480-520 mls/min 380-420 mls/min
1X Items (large)	Items larger than 3 x 3 x 3 feet. Items should be placed on a pallet and wrapped in plastic. For extremely large items 2 samples should be taken on the item.	1 hour	400 mls/ min	380-420 mls/min
Perimeter Monitoring for Clean-up Sites	8 perimeter positions shall be set up. Dual tubes shall be used for all positions. Perimeter positions shall not exceed 60 x 60 meters.	2 hours 4 hours	200 mls/min 100 mls/min	190-210 mls/min 100-110 mls/min
Shelter and Position A	Positions located at the shelters shall be located at the point source.	4 hours	500 mls/min	480-520 mls/min

Figure-A1 (Flow Table)

A1.0 Air Sample Record Sheet

1. All pertinent sample data shall be recorded on the scratchlog and collected in Bldg E-3344. All data from off-site operations will be collected on a TAG computer and will be downloaded and added to the data in the main computer upon the return to the Monitoring Branch.

2. The following required information is recorded in the appropriate location of the scratchlog or the flow log.

(1) Scratchlog (MBFORM-12)
- (a) Sample number (assigned by computer)
- (b) Building or off-site location **(LDCK)**
- (c) Sample identification number **(See Figure-5 for perimeter ID)**
- (d) Date of sample **(date sample is taken)**
- (e) Agent(s) being tested for **(HD/L only)**
- (f) Type of sample **(Background, TAPC, XXX or SOIL.)**
- (g) Time ON and OFF for samples
- (h) Point of contact and extension number **(Sanders, 410-676-1395)**

(2) Air Monitoring Worksheet (MBFORM-39)
- (a) Sample number
- (b) Building or location
- (c) Sample identification (Item/Grid ID)
- (d) DAAMS tube numbers (lowest number listed first)
- (e) Flow rates for all tubes prior to sample being run

*Field Tags/Labels are used interchangeably in this document

(f) Flow rates for all tubes at conclusion of sample run
(g) Comments on any discrepancies concerning tube flows
(i.e. tubes contaminated with dirt/liquid, tube broken).

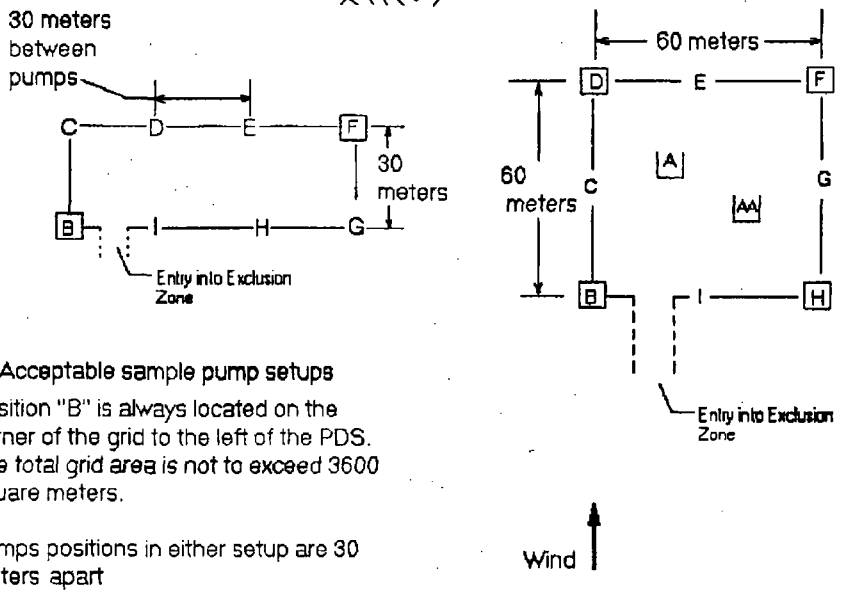
A2.0 Perimeter Monitoring

Perimeter monitoring will be used for historical purposes. As there is a constant fluctuation in the wind direction, an array of eight pumps will be utilized to provide the working personnel and general community near the working area with maximum monitoring. Sample results are legal documentation of worker exposure. Copies of personnel air sampling results that are above the permissible limits will be forwarded to the Edgewood Clinic for inclusion in ECBC employee medical records. All personnel air sampling results, both positive and negative, will be made available to the Clinic upon request. Maintain all sampling records per AR 40-5 requirements.

A2.1 Set up

a. Refer to section 2.0 of MT-11 sampling equipment set up.

b. Location of perimeter pumps is noted in Figure 4. Perimeter pumps will be moved collectively as the work area progresses forward, but the overall work area will not exceed total area of 3600 square meters. Distance between Sample pumps will not exceed 30 meters, with the exception of the "A" pumps within the grid. Technician will ask the Site Safety Officer where the PDS will be located in order to determine the location of pump position "B". Pump positions are place clockwise once position "B" is determined.



Acceptable sample pump setups

Position "B" is always located on the corner of the grid to the left of the PDS. The total grid area is not to exceed 3600 square meters.

Pumps positions in either setup are 30 meters apart

Figure-A2 (Perimeter pump Locations)

*Field Tags/Labels are used interchangeably in this document

A2.2 Air Sample Record Sheet.

Per Memorandum received from the Environmental Conservation and Restoration Division (ATTCH-t 1) - Upon completion of the sampling period, check with the MINICAMS to see if any readings were obtained for HD/L. If no readings occurred, recondition all DAAMS and reset the pumps. No samples need to be entered into the sample database. If any readings for HD or L are obtained - **Then flow rates for all tubes will be recorded and positions entered into the database**. All positions entered into the database will contain the information noted in Figure-5.

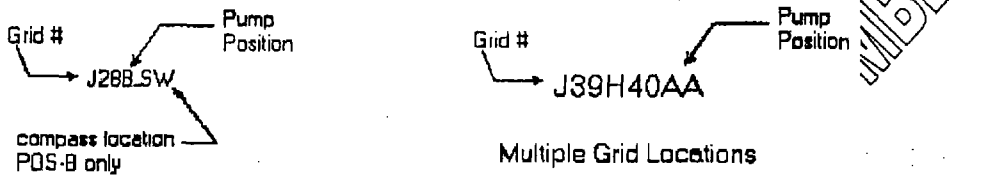


Figure-A3 (Position Identification)

2.4 Notification.

A2.3 Air Sampling Records.

Send copies of air sampling results per the instruction of paragraph 3.0 of MT-11.

*Field Tags/Labels are used interchangeably in this document

MT-11, Appendix B: EDS

B1.0 EDS Setup

All equipment setup and monitoring for the EDS site will be in accordance with procedures indicated in MT-11. Specific flow rates and time intervals for EDS sampling procedures are indicated in Figure-B2 of this appendix. The set up of the EDS structure is indicated in Figure-B1. The position of the MINICAMS and the filters may vary slightly from site to site. The DAAMS positions shall be labeled as shown in diagram.

EDS Layout

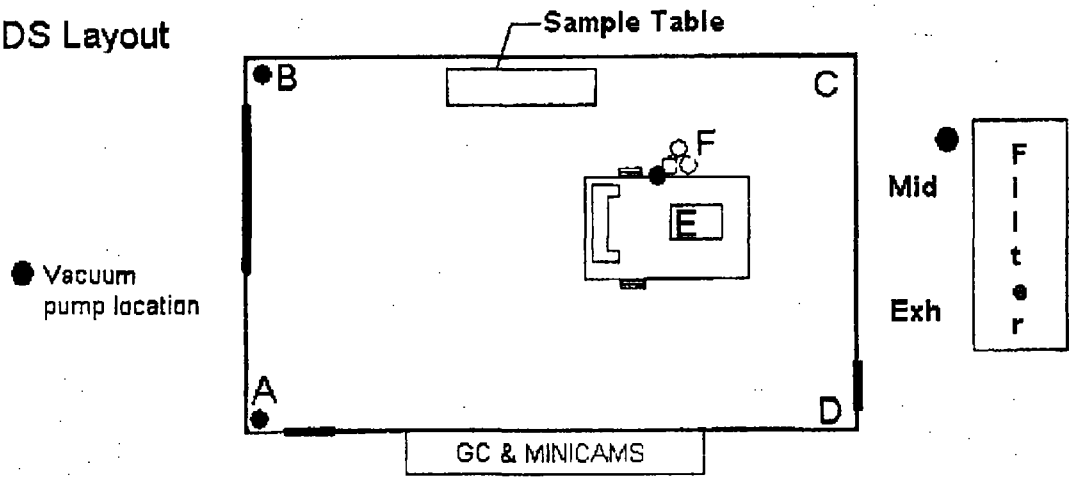


Figure-B1

B1.1 Sample Intervals for EDS

1. Positions shall be identified as indicated in (1) through (4). All labels will be written and entered the same with no extra spaces, dashes etc.
   (1) A; B; C and D shall be labeled POS.A; POS.B; POS.C and POS.D
   (2) E shall be identified as EDS
   (3) F shall be identified as WASTE DRUMS
   (4) Filter positions shall be labeled MID (for Mid bed) and EXH (for Exhaust).

B1.2 Sampling times and flow rates are identified in Figure-B2.

(1) Allowable times, flows and limitations are noted in Figure-6. DO NOT EXCEED noted flow or time limitations

(2) NOTE: **There are no lower limits for flow rates 100 mls or below.**

Monitoring Flow and Sample Rates Dual DAAMS tubes shall be used for all monitoring				
Sample Type	**Description and preparation procedures prior to sampling**	**Sample Time**	**Target Flow Rate**	**Acceptable Flow Limits**
Background Monitoring	Positions A - D set in a clockwise pattern.	4 hours 8 hours	100 mls/min 050 mls/min	100 - 110mls/min 50 – 55 mls/min
Confirmation Monitoring	WASTE DRUM and EDS positions. Sample period is NTE 12 hours.	NTE 12 hours	500 mls/min	480-520 mls/min
Filter Monitoring	Filter positions are NTE 12 hours. DAAMS tubes are pulled after 3rd consecutive MINICAMS alarm. Flows are to be taken.	NTE 12 hours	500 mls/min	480-520 mls/min

Figure-B1

*Field Tags/Labels are used interchangeably in this document

MT-11, APPENDIX C: UK Donovon Site

UK Donovon Setup

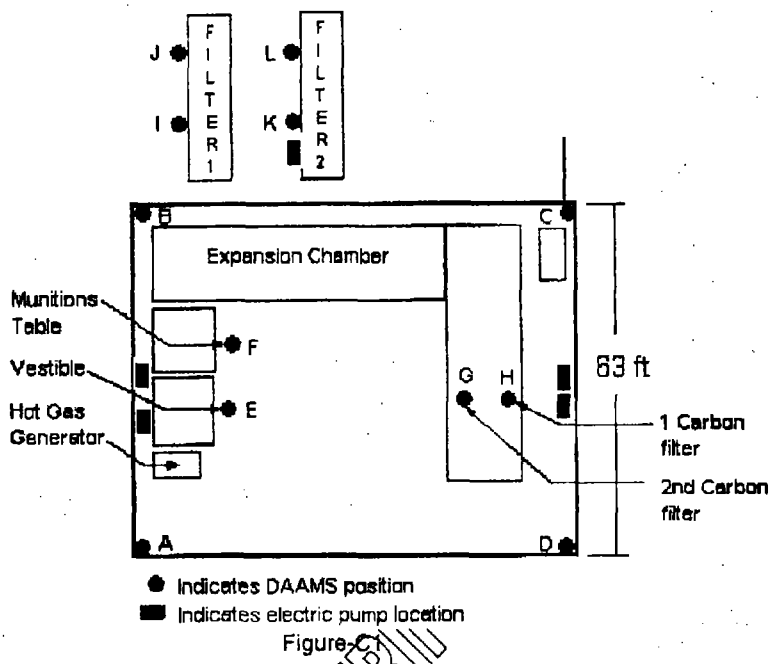


Figure-C1

C1.1 Perimeter Positions for Donovon Chamber (see Figure-C1).

2. Positions shall be identified as indicated in (1) through (4). All labels will be written and entered the same with no extra spaces, dashes etc.
   (1) A; B; C and D shall be labeled POS.A; POS.B; POS.C and POS.D
   (2) E shall be identified as Vestible
   (3) F shall be identified as Munitions
   (4) G and H positions shall be labeled Carbon 1 and Carbon2.
   (5) Filter positions shall be labeled MID (for Mid bed) and EXH (for Exhaust).

C1.2 Sampling times and flow rates are identified in Figure-C2.

(1) Allowable times, flows and limitations are noted in Figure-6. DO NOT EXCEED noted flow or time limitations

(2) **NOTE: There are no lower limits for flow rates 100 mls or below.**

Monitoring Flow and Sample Rates Dual DAAMS tubes shall be used for all monitoring				
Sample Type	**Description and preparation procedures prior to sampling**	**Sample Time**	**Target Flow Rate**	**Acceptable Flow Limits**
Perimeter - Monitoring	Positions A - D set in a clockwise pattern.	4 hours 8 hours	100 mls/min 050 mls/min	100 - 110mls/min 50 – 55 mls/min
Confirmation Monitoring	Vestibule, Munitions table, Carbon filter 1 and 2 time intervals are NTE 12hours. DAAMS tubes are pulled after 3rd consecutive MINICAMS alarm. Flows are to be taken.	NTE 12 hours	500 mls/min	480-520 mls/min
Filter Monitoring	Filter positions are 8 hours.	8 hours	50mls/min	100-110 mls/min

*Field Tags/Labels are used interchangeably in this document

Figure-C2

C1.0 Air Sample Record Sheet

1. All pertinent sample data shall be recorded on the scratchlog and collected in Bldg E-3344. All data from off-site operations will be collected on a Sample Team computer and will be downloaded and added to the data in the main computer upon the return to the Monitoring Branch. Data shall be filed in the following sequence – Scratchlog, followed by Flowsheets with Datasheets filed last. Documents shall be filed numerically by year, month and sample identification number.

2. The following required information is recorded in the appropriate location of the scratchlog or the flow log.

(2) Scratchlog (MBFORM-12)
- (a) Sample number (assigned by computer)
- (b) Building or off-site location **(UDON)**
- (c) Sample identification number **(See Figure-C1 for position IDs)**
- (d) Date of sample **(date sample is taken)**
- (e) Agent(s) being tested for **(HD/L only)**
- (f) Type of sample **(Background, TAPC, XXX or SOIL.)**
- (g) Time ON and OFF for samples
- (h) Point of contact and extension number **(DIBerardo, 410-436-3103)**

(2) Air Monitoring Worksheet (MBFORM-39)
- (a) Sample number
- (b) Building or location
- (c) Sample identification (Item ID)
- (d) DAAMS tube numbers (lowest number listed first)
- (e) Flow rates for all tubes prior to sample being run. See Figure-C2 for allowable flow rates and limitations
- (f) Flow rates for all tubes at conclusion of sample run
- (g) Comments on any discrepancies concerning tube flows (i.e. tubes contaminated with dirt/liquid, tube broken).

*Field Tags/Labels are used interchangeably in this document

APPENDIX D: Pine Bluff Arsenal

All of the procedures and equipment settings noted in the main body of this IOP apply to this appendix. Specific monitoring requirements for the project are outlined in this appendix.

D1.1 DAAMS Positions for Pine Bluff site (see Figure-D1).

1. Positions shall be identified as indicated on the site diagram. All labels will be written and entered as indicated with no extra spaces, dashes etc.

2. Individual locations identifications are listed below. The 3-letter identifier is to be used when entering sample information into the Sample database. Filter positions shall be labeled PBA.MID (for Mid bed) and PBA.EXH (for Exhaust). DO NOT make changes/adjustments.

IAL - Inlet Airlock
UPR - Unpack/pack Room
OAL - Outlet Airlock
DZ - Drop Zone
DCR - Decon Room
PPE - PPE Drop Room
SCB - SBA Drop Room

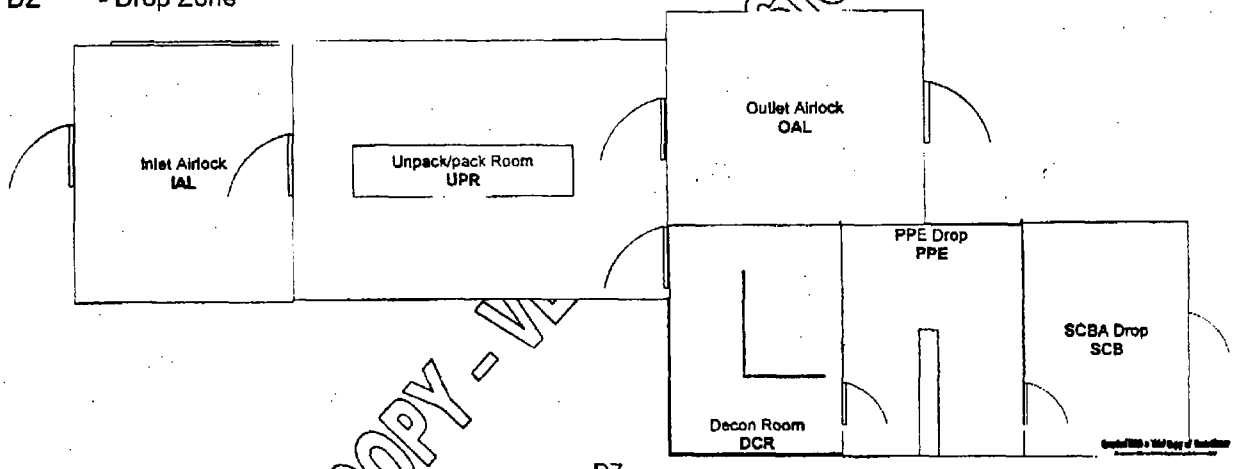


Figure D-1

D1.2 Sampling times and flow rates are identified in Figure-D2.

1. Allowable times, flows and limitations are noted in Figure-D2. DO NOT EXCEED noted flow or time limitations
2. **NOTE: There are no lower limits for flow rates 100 mls or below.**

Monitoring Flow and Sample Rates
Dual DAAMS tubes shall be used for all monitoring

Sample Type	Description	Sample Time	Target Flow Rate	Allowable Flow Limits
XXX and TAPC	Equipment/Clothing generated at the site requiring clearances before re-using	1 hour 48 min.	400 mls/min 500 mls/min	380-420 mls/min 480-520 mls/min
Confirmation Monitoring	Unloading area(DZ) intervals are NTE 12hours. DAAMS tubes are pulled after 3rd consecutive MINICAMS alarm. Flows are to be taken.	NTE 12 hours	500 mls/min	480-520 mls/min
Filter Monitoring	Filter positions (PBA.MID & PBA.EXH) are NTE 12hours.	NTE 12 hours	500mls/min	480-520 mls/min
Background Monitoring	Sample points located within steel structure	8 hours	50 mls/min	50 – 55 mls/min

*Field Tags/Labels are used interchangeably in this document

Figure-D2

D1.0 Air Sample Record Sheet

1. All pertinent sample data shall be recorded on the scratchlog and collected in Bldg E-3344. All data from off-site operations will be collected on a Sample Team computer and will be downloaded and added to the data in the main computer upon the return to the Monitoring Branch. Data shall be filed in the following sequence – Scratchlog, followed by Flowsheets with Datasheets filed last. Documents shall be filed numerically by year, month and sample identification number.

2. The following required information is recorded in the appropriate location of the scratchlog or the flow log.

(3) Scratchlog (MBFORM-12)
- (a) Sample number (assigned by computer)
- (b) Building or off-site location **(PBA)**
- (c) Sample identification number **(See Figure-D1 for position IDs)**
- (d) Date of sample **(date sample is taken)**
- (e) Agent(s) being tested for **(HD/L only)**
- (f) Type of sample **(Background, TAPC, XXX or SOIL.)**
- (g) Time ON and OFF for samples
- (h) Point of contact and extension number **(D. Bolt 410-436-5903)**

(2) Air Monitoring Worksheet (MBFORM-39)
- (a) Sample number
- (b) Building or location
- (c) Sample identification (Item ID)
- (d) DAAMS tube numbers (lowest number listed first)
- (e) Flow rates for all tubes prior to sample being run. See Figure-D2 for allowable flow rates and limitations
- (f) Flow rates for all tubes at conclusion of sample run
- (g) Comments on any discrepancies concerning tube flows (i.e. tubes contaminated with dirt/liquid, tube broken).

D2.0 Labwaste/XXX/TAPC items

Labwaste is any trash generated during agent operations that requires monitoring before disposal.

XXX items are equipment or tools that have been used or potentially exposed to agent during agent operations

TAPC is clothing or items that will be worn again. This includes reusable boots, gloves, coveralls, masks, respirators, uniforms, SCBA and associated equipment/harnesses. It does not include disposable coveralls, booties or gloves.

D2.1 All of the above items must be double-bagged, dry and sealed for a minimum of 4 hours at an ambient temperature of 70 degrees Fahrenheit.

D2.2 **Gloves will be worn** when sampling any of the above items. Dispose of gloves appropriately at conclusion of samples.

D2.3 Refer to section 2.0 of this IOP for equipment setup procedures.

D2.4 Barcodes affixed to items will be used as the sample identification. If no barcode is present, the ST will assign an item identification number in the following format (PBymmdd###;

*Field Tags/Labels are used interchangeably in this document

example PB20201001). The format indicates the Site Location, year, month, day and the numerical sequence of the item.

D2.5 Allowable times, flows and limitations must NOT EXCEED noted flow or time limitations. See Figure-D3.

Sample Type	Description	Time	Target Flow	Allowable Flow Limits
Labwaste/XXX or TAPC items	PPE or trash generated during agent operations	48 min 1 hour	500 mls/min 400 mls/min	480 – 520 mls/min 380 – 420 mls/min

Figure-D3

*Field Tags/Labels are used interchangeably in this document

Attachment 5

ECBC Internal Operating Procedures MT-13 and MT-16

Internal Operating Procedure
Operations Directorate Monitoring Branch

Title: Analysis of Chemical Warfare Agents and Degradation Products on DAAMS Tubes using Gas Chromatography System Coupled with a Mass Spectrometer Detector (GC/MS)

Division: Chemical Operations

Branch: Monitoring Branch

Building/Area: E3330 and other areas

This internal operating procedure covers operations, methods and procedures of a general nature not covered by a standing operating procedure. This procedure will be effective until rescinded or superseded. Changes to this procedure will be accomplished by submission of revisions or amendments for approval. This IOP meets "Written Method" requirements of Revision 4 of the CASARM QA Plan.

Originator: Gail M. Soubie Date: 7/7/03
Gail M. Soubie

Analytical Team Leader: Steven D. Norman Date: 6/30/03
Steven D. Norman

Quality Assurance Coordinator: Paul L. Stewart Date: 6/30/03
Paul L. Stewart

Monitoring Branch Chief: Thomas E. Rosso Date: 30 June 03
Thomas E. Rosso

Date: June 2003
IOP Number: MT-13
Revision Number: 6
Prepared by: G.M. Soubie (ext. 5-3424)
Revisions by: C.V. Giannaras (ext. 5-2520)
Approved by: T.E. Rosso (ext. 5-8428)

RECORD OF CHANGE

Previous revisions of this document did not incorporate a record of change page.

- Revision 6 – IOP was altered by removing references to GCFPD DAAMS analysis. For GCFPD analysis, see IOP MT-19.
- Revision 6 – Incorporated changes required by CASARM QA Plan, Revision 4.
- Revision 6 – Incorporated the following laboratory changes: surrogate added to all samples and QC analyses, removed requirement of a QL every 10 samples, removed requirement of a new calibration every fourteen days, instrument tuning via injection of tuning solution (BFB).

Table Of Contents

Title: Analysis of Chemical Warfare Agents on DAAMS using Gas Chromatography System Coupled with a Mass Spectrometer Detector (GC/MS)

Operator's Statement: I have read, or have had read to me, the procedures in this IOP. I, by my signature below, indicate that I thoroughly understand and agree to abide by these instructions.

Name (print)	Signature	Date

Supervisor's Statement: I have personally reviewed this IOP and, to the best of my knowledge, believe that the information listed herein is correct.

Supervisor:_____ Date:_____
Thomas E. Rosso

Analysis of Chemical Warfare Agents on DAAMS using Gas Chromatography System Coupled with a Mass Spectrometer Detector (GC/MS)

1.0 GENERAL INFORMATION

1.1 The Gas Chromatography (GC) System coupled with a Mass Selective Detector (MSD) can detect chemical warfare (CW) agents GA, GB, GD, GF, HD, HN-1, HN-3, L (derivatized with BME), and VX (as G-analog). The CW agents are introduced into the GC system by use of the Depot Area Agent Monitoring System (DAAMS) solid sorbent tubes. The ACEM 900 thermal desorbtion system will desorb the agents from the DAAMS tubes onto an analytical column in the GC that separates the agents and then these agents pass to the MSD.

1.2 The MSD may be used for confirmation or initial analysis. The MSD is a very specific detector that breaks each compound down into ion fragments specific to that compound. This allows both qualitative and quantitative analyses. Peak identification is based upon retention time comparison with external standard calibration and by evaluation of the unique spectrum.

1.3 The DAAMS tube is filled with sorbent material, which traps the agents from air flowing through the tube. The DAAMS tube is designed to collect and trap semi-volatile organic compounds, which are released during thermal desorption using an ACEM desorber. A specific flow rate and sampling time are used to collect each DAAMS sample and each sampling location consists of two DAAMS, one for analysis and one for confirmation.

1.4 The ACEM desorber will go through tube dry, tube heat, tube cool, trap heat and system recycle steps. During the tube heat step, the tube is heated, transferring the collected sample to a focusing trap present in the ACEM. During the trap heat step the focusing trap is heated, injecting the collected sample onto the GC column. At the completion of the system recycle step the ACEM is ready to accept the next tube for analysis.

2.0 Data Collection and Storage

2.1 The data collected from each GC are stored electronically on a Laboratory Information Management System (LIMS) and/or Chemstation. These electronic data and the paper hardcopy chromatograms serve as historical records and are stored in a safe storage place. The chromatogram consists of peak area, retention time, and other related information. Results of analyses are checked for validity. All data is reviewed by the Laboratory Manager and/or designated Senior Staff.

2.2 The electronic systems report the results in nanograms (ng). These values are evaluated with reference to sampling time and rate of airflow during sampling (i.e. AEL level for GB is 7.2 ng if sampled for 120 minutes at 0.6 Liters per minute airflow). Results are electronically transferred on the data sheet (see Appendix IV) supplied by the LIMS system containing information about sampling site, flow rate, sampling time etc. Result of analysis is conveyed to the Point of Contact (POC).

3.0 CALIBRATION PROCEDURES

3.1 The GCs shall be recalibrated in the following events: the GCs or individual components have undergone extensive repair, the instrument is out of control, or at other times when the operators deem it necessary. Instructions listed in section 3.5 shall be followed for recalibration.

3.2 Separate standards will be prepared for calibration and QC checks and will be used for calibration and for QC samples (i.e. use calibration standards to calibrate the instrument and use the QC standards, QLs and QPs, to check instrument operation).

> *NOTE: All calibration standards, QLs, QPs, and samples analyzed shall be fortified with a surrogate compound and evaluated in accordance with Appendix II.*

3.3 Preparation of calibration standards

3.3.1 Remove agent calibration standard and QC standard from cold storage and place in fume hood. Allow standard to reach room temperature.

3.3.2 Using a 10ul syringe, withdraw the required amount of agent from the QC standard solution vial(s). Spike the tube in the spiking assembly by inserting the syringe needle into the DAAMS tube and depressing the plunger on the syringe. Allow the spiked tube to aspirate for 1 minute. If analyzing for Lewisite (L), spike the tube with 5ul of 0.05%BME. See Section 8.0 for V-G pad analysis.

3.3.3 Remove the spiked calibration tube from the spiking assembly after one minute and place into the ACEM tube desorber.

3.4 Tune Verification

3.4.1 At the beginning of each operational day, the analyst will perform a tune verification on the GC/MSD in accordance with requirements specified in Appendix III.

3.4.2 If operations exceed 12 hours, another tune verification shall be performed and evaluated. Upon completion of the tune verification a QL shall be analyzed to ensure that instrument is in control.

3.5 Initial Calibration

3.5.1 Calibration must be performed using the sample introduction technique that will be used for samples.

3.5.2 Calibration of an analytical instrument involves the delineation of the relationship between the response of the instrument and the amount or concentration of an analyte introduced into the instrument. The graphical depiction of this relationship is often referred to as the calibration curve. In order to perform quantitative measurements, this relationship must be established prior to the analysis of any samples, and thus, is termed initial calibration. The initial calibration for this

chromatographic method involves the analysis of standards containing the target compounds at a minimum of three different concentrations covering the working range of the instrument

3.5.3 At a minimum, a three-point calibration curve is required. Additional points may be analyzed if necessary. The typical standards used are "air combo" in hexane, "Lewisite" in methyl alcohol, and the surrogate solution in hexane. Typical concentrations of the standards are listed in the Table 1. Each standard is spiked separately onto the DAAMS tube on the spiking block under a flow of 100 ml/minute Nitrogen with the exception of VX-G-Analog, which is aspirated through the V-G pad (see Section 8.0). If monitoring for lewisite, spike 5ul of 0.05% BME, 1 ul, 3 ul, and 7 ul of each calibration standard solution are used for each calibration level. (These amounts are equivalent to 0.25, 0.75, and 1.75 AEL, except for the vesicants which have a considerable higher AEL). Flow is maintained on the DAAMS tube for about one minute after it is spiked with the standards.

3.5.4 Tabulate the area response against the concentration for each target analyte.

3.5.5 Linear calibration using a least squares regression

3.5.5.1 The analyst must employ a linear regression equation that does not pass through the origin for all surrogates and agents. This is most easily achieved by performing a linear regression of the instrument response versus the concentration of the standards. Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). This is a statistical requirement and is not simply a graphical convention. For all instances, an appropriate unweighted least squares method should be used. The regression will produce the slope and intercept terms for a linear equation in the form:

$y = ax + b$

where:
y = instrument response (peak area or height)
x = concentration of the calibration standard
a = slope of the line (also called the coefficient of x)
b = the intercept

The analyst must not force the line through the origin, but have the intercept calculated from the data points. The use of a linear regression may not be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards. The regression calculation will generate a correlation coefficient (r^2) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.000 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.990. In calculating sample concentrations by the external standard method, the regression equation is rearranged to solve for the concentration (x), as shown below.

$x = (y-b)/a$

If the correlation coefficient does not meet criteria, up to two points on the curve may be re-shot to bring the value up to criteria. If criteria still aren't met, the entire curve must be re-shot. (The two re-shots as above may be included in the new curve.)

3.6 Initial Calibration Verification

3.6.1 Immediately following the initial calibration, a calibration verification QL must be performed. This is accomplished by analyzing a 3ul QL. The results from the calibration standard analysis should meet the verification acceptance criteria provided in Sec. 3.7.1.1.

3.6.2 The calibration verification spiking standard must not be prepared from the same standards as the initial calibration standards.

3.6.3 If the initial calibration verification QL fails, corrective actions shall be performed on the instrument and documented on the Excel® Daily Run Log comments section and the Excel® QC Chart comments section. The instrument must be recalibrated as described in Section 3.5.

3.6.4 Instrument maintenance and changes to system parameters as part of the corrective action must be recorded in the instrument logbook and initialed and dated.

3.7 Calibration Verification (On-going Calibration)

3.7.1 The calibration curve (Sec. 3.5) for each compound of interest must be verified at least once every 12 hours prior to sample analysis and at the end of sample analysis, using the introduction technique used for samples. This is accomplished by analyzing a 3ul QL prepared with the QC standard. Additional QLs during sample analysis is strongly recommended (i.e., after every 10 sample injections). All samples must be bracketed by QLs. The results from the QC standard analysis must meet the following verification acceptance criteria:

3.7.1.1 If the percent difference between the found and true amount for any target agent is less than or equal to +/-15%, the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater than 15% difference between the found and the true amount), for any one agent, then corrective action must be taken prior to the analysis of samples. If the agent(s) not meeting the +/-15% criteria are not included in the list of analytes for a sampling event, no corrective action is required. If the first QL of the day fails this +/-15% test, corrective actions shall be performed on the instrument and documented on the Excel® Daily Run Log comments section and the Excel® QC Chart comments section. The instrument must be recalibrated as described in Section 3.5. Any time an instrument is calibrated, new QLs must be analyzed and quantitated against the new calibration curve. If any target agent in an ongoing or ending QL exceeds the +/-15% criteria, then the system is considered out-of-control and analysis must cease. See Section 7.0 for out-of-control situations.

3.7.2 The calibration verification spiking standard must not be prepared from the same standards as the initial calibration standards.

3.7.3 Instrument maintenance and changes to system parameters as part of the corrective action must be recorded in the instrument logbook and initialed and dated.

4.0 **Blank analysis**

4.1 Place a pre-conditioned DAAMS tube into the ACEM desorber and depress the "Start" button. Review the chromatogram for instrument contamination. If the blank contains interference peaks,

run additional blanks until no interferences are detected. A method blank should be analyzed immediately before the analysis of QPs and samples, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to bake the system for an extended period of time.

5.0 Procedure

5.1 Receipt of Samples

5.1.1 DAAMS samples are received from the Sample Collection Team (SCT) after they have been logged into the Horizon Laboratory Information Management System (LIMS). The scratch log is signed to demonstrate chain-of-custody. The signed scratch log is returned to the SCT technician. Samples not analyzed the same day as collected are stored in the sample refrigerator at 4 degrees Celsius until analyzed.

5.1.2 For field operations, DAAMS samples are logged into the TAG program that generates the scratch log. The samples are then delivered to the MAP for analysis. The scratch log is signed by the analyst to demonstrate chain-of-custody.

5.2 Batch assignment in LIMS (Horizon)

5.2.1 The analyst assigns samples to a batch in the LIMS before or during instrumental analysis. Samples and QPs assigned to a batch shall be sampling event specific (see Section 6.1.3). A batch is a group of samples unique to a sampling event and analyzed on a given instrument on a given day that includes QP samples collected with the samples being analyzed and QL samples. The analyst logs QPs and samples into the LIMS batch. The analyst adds QLs to the batch as they are analyzed. There must be at least two QLs analyzed per instrument per day and one QP per twenty samples. All calibration analyses shall be added to the batch when the instrument is calibrated. The analytical batch size is limited to twenty samples and their associated QC samples. Only one acceptable QP can be contained in a LIMS batch. If more than one QP was analyzed before an acceptable QP achieved, the failing QP(s) must be flagged in LIMS as "XX". Currently, LIMS can only have one QP in a batch therefore every analytical batch must be in a separate LIMS batch and not exceed 20 samples.

5.3 Field operations

5.3.1 DAAMS samples are received from the Sample Collection Team (SCT) and the samples are analyzed by on-site MAP analytical equipment or shipped to ECBC for analysis in accordance with approved site procedures. Analytical results are reported as required by the approved site procedures.

5.3.2 QL and QP acceptance requirements shall be the same as ECBC laboratory acceptance requirements. A minimum of 2 acceptable QLs per day and one QP per twenty samples shall be required for each day of operation for each instrument and each agent of interest. See sections 3.6.1.1 (QL) and 6.1.5.1 (QP) for specific requirements. All samples shall be bracketed by QLs.

5.3.3 The sample technician enters the analysis results in the TAG program. If a sample is entered as "not clear", the TAG program will permit the 2nd tube to be logged in and analyzed.

5.4 Sample Analysis

5.4.1 Initial Analysis

5.4.1.1 The DAAMS tube will be placed in the ACEM 900 for thermal desorption and subsequent analysis by gas chromatography. The analyst will review the data and determine whether the sample is clear or not clear for the agents of interest by evaluating the extracted ion chromatograms.

5.4.2 Confirmation Analyses

5.4.2.1 Samples received from the GC/FPD laboratory for confirmation will have the second tube analyzed via GC/MSD. The sample ID number as well as the compound of interest must be written on the label.

5.4.2.2 The second DAAMS tube will be placed in the ACEM 900 for thermal desorption and subsequent analysis by gas chromatography. The analyst will review the data and determine whether the sample is clear or not clear for the agents of interest by evaluating the extracted ion chromatograms.

5.4.2.3 The analyst will indicate on the quantitation report that the analysis was for confirmation.

5.5 Re-conditioning of DAAMS Tubes

5.5.1 All DAAMS tubes analyzed and tubes not analyzed will be re-conditioned after analysis is completed, documented and results reported.

6.0 Quality Control

6.1 QP Preparation and Analysis

6.1.1 The function of the Quality Process (QP) is to correct results for method recovery. Positive sample results are corrected by this method recovery as directed in the Monitoring Branch Quality Control Plan.

6.1.2 The QP DAAMS tubes are aspirated at the appropriate sampling site for the required amount of time and subsequently returned to the laboratory or Mobile Analytical Platform (MAP) for analysis. A minimum of two QPs shall accompany every sample set. More QPs may be included to assure a minimum of one acceptable QP sample per 20 samples.

6.1.3 A sample set is defined as samples of the same sampling event analyzed each day of operation (i.e., samples from J-Field, 5100, or Shotglass).

NOTE: For purposes of this document, labwaste samples in a defined area will be considered a specific sampling event. For example, if 13 labwaste bags are located

in Building 5100, a QP is necessary for those 13 samples, not each individual bag. Also, if there are two or more locations with labwaste samples, each location will have an associated QP. For any QP monitoring for lewisite, spike 5ul of 0.1% BME.

6.1.4 If there is a need to prepare QPs in the laboratory, connect the glass frit end of the tube to the spiking assembly. Spike the tube in the spiking block with 4ul of the appropriate QC standard(s). See Section 8.0 for spiking onto a V-G pad. If monitoring for lewisite, spike 5ul of 0.05% BME. Attach the glass wool end of the spiked tube to the vacuum pump. Set the vacuum flow to be 0.25 liters per minute or an equivalent sample volume. Record the date, on time, and flow rate on MBFORM-47. Run the system for 96 minutes at this flow rate. After the 96 minutes has elapsed, measure the flow through the tubes and record off time and ending flow rate on MBFORM-47. Remove the tubes and transfer to the ACEM for analysis.

6.1.5 QP samples will be introduced during the analysis routine at one QP per 20 samples. This QP sample validates the ability to detect the agent in field samples. QPs will be spiked using the QC standard(s) and will be of the same sampling event as the samples being analyzed. Until field QP limits are established, a minimum percent recovery of 10% must be achieved as long as the instrument is shown to be operating properly as shown by acceptable QLs.

6.1.5.1 The QP must have a recovery within the laboratory established limits for each agent (see Section 10.0). If a QP response falls above the upper established laboratory limit, a second QP must be analyzed. If the second QP response fails high, the QP with the lower response will be used with the batch of associated samples and the analyst will note in the comments section the failure. These failures will be closely monitored for trends. Both QPs will be entered in the LIMS batch with the QP not being used flagged as "XX".

6.1.5.2 If a QP response falls below the lower established laboratory limit, a second QP must be analyzed. If the second QP response is within established laboratory QC limits, analysis may proceed of the samples associated with that QP. If the second QP response falls below the lower established laboratory QC limit, analyze the remaining QPs associated with the samples until one is within established laboratory QC limits. If one QP falls within established laboratory QC limits, analysis may proceed of the samples associated with that QP. If none of the associated QPs are within the established laboratory QC limits, all analyses must cease for all samples associated with those QPs. Those samples will need to be re-sampled. All QPs will be entered in the LIMS batch with the QPs not being used flagged as "XX".

NOTE: In case of a unique sample, one that can not be re-sampled, analysis may proceed without a passing QP. This will be determined on a case-by-case basis by the Laboratory Manager or Branch Chief. If the samples are analyzed, it must be noted on the Daily Run Log, entered in LIMS, and communicated to the client that the QP failed and results are not defensible.

6.1.6 Only one sampling event will be put in a LIMS batch. For samples received without QPs, a laboratory prepared QP shall be used. Samples received without QPs will be treated as a single sampling event, therefore samples from different sites may be associated with the same laboratory QP. Different sampling events with QPs must not be entered into the same LIMS batch.

6.1.7 Positive results for sample analyses must be corrected by the associated QP recovery (only when the QP recovery is less than 100%). The LIMS (Horizon) does this automatically.

7.0 Out-of-Control Situations

7.1 Instrument Control

7.1.1 If one or more agents of a multiple agent QL is less than the -15% requirement (i.e., recovery is low) the instrument is out of control. Samples analyzed after the last in-control QL are considered suspect and must be reanalyzed for the failing agent(s). Corrective action must be performed and documented before continuing any analyses. Sample analysis shall not continue until the system is shown to be in control.

7.1.2 If one or more agents of a multiple agent QL is greater than the +15% requirement (i.e., recovery is high) but less than +50% (150%), the instrument is out of control. The analytical results of all samples analyzed since the last in-control QL that do not exceed 1 AEL can be cleared. If any sample has a target agent(s) detected above 1 AEL and that agent(s) fails the QC criterion, the sample shall be re-analyzed and/or re-sampled for the failing agent(s). Sample analysis shall not continue until the system is shown to be in control.

7.1.3 If the QL response is greater than +50% (150%) of the target concentration, the instrument is out of control. All samples back to the last acceptable QL are suspect and these samples shall be re-analyzed and/or re-sampled for the failing agent(s). Corrective action must be performed and documented before continuing any analyses. Sample analysis shall not continue until the system is shown to be in control.

7.1.4 All actions shall be documented on the Excel® Daily Run Log comments section and the Excel® QC Chart comments section.

7.2 Statistical Control

7.2.1 Out-of-statistical-control situations are defined by the placement and trends of daily average and range QL results on the control charts. An out-of-statistical-control situation does not call for an immediate cessation of analysis. It is an indication that the system could go out of control. The criteria for out-of-statistical-control situations are as follows:

7.2.1.1 A daily QL average that is outside the control limits on the x-bar (average) chart.

7.2.1.2 A point that is above the upper limit on the R (range) chart.

7.2.1.3 Eight (8) or more successive points on the same side of the central line on the x-bar (average) chart or R (chart).

7.2.2 If an out-of-statistical-control situation occurs, the analyst shall examine the system for unusual conditions and may perform several routine maintenance procedures in an attempt to determine the cause of the out-of-statistical-control situation. Re-calibration may be required.

7.2.3 All actions shall be documented on the Excel® Daily Run Log comments section and the Excel® QC Chart comments section. All instrument maintenance shall be documented in the instrument logbook.

8.0 Analysis of G-analog of VX

8.1 VX is converted to it's G-analog by incorporating a silver floride pad. All calibration and QC standards shall be prepared using this technique for samples that were collected via the silver floride pad.

8.2 For G-analog of VX, attach a silver floride pad (V-G pad) on the frit end of the DAAMS tube. This pad is placed inside a Teflon® Swagelok® fitting which is fitted to the sampling end of the DAAMS tube. Place the syringe needle on the top of the V-G pad and depress the plunger, allowing the VX standard to be aspirated through the V-G pad onto the DAAMS tube by using a vacuum pump for 1 – 2 minutes.

8.3 Each silver floride pad should be replaced monthly or as needed. Typically, in lab use of a silver floride pad will allow for a longer effectiveness compared to pads used in the field.

9.0 Data Review

9.1 At the end of each day, the analyst reviews his/her batches, prints the Excel® control charts, completes the checklist (MBFORM-50), initials and dates the daily report to indicate that the data are correct, and prints the data summary report from LIMS. These reports are attached to the data for review by the Laboratory Manager or designated senior staff.

9.2 Designated senior staff will review the data, initial the daily report as being acceptable and then will perform the management review function in the LIMS system.

9.3 The QA officer will review the daily report and perform the QA review function in the LIMS system.

9.4 The Monitoring Branch Chief and the Point-of-Contact (POC) will be informed as soon as possible after a confirmed result above AEL (TWA) is noted.

9.5 The required action levels for DAAMS tube calculations are listed in the Monitoring Branch Quality Control Plan and have been included in this document for convenience (see Appendix I and Table 2).

10.0 QL and QP Limits

10.1 QL and QP control chart limits will be generated from historic data per agent per instrument when acceptable data is available. These limits will be monitored regularly and updated at a minimum yearly. More frequent updates may be needed to account for seasonal changes.

10.2 The QL and QP limits are calculated using the following statistical formula:

- Upper Control Limit = $UCL_{\overline{X}} = \overline{\overline{X}} + \overline{R}(1.880)$
- Central Line = $\overline{\overline{X}}$
- Lower Control Limit = $LCL_{\overline{X}} = \overline{\overline{X}} - \overline{R}(1.880)$

11.0 HAZARDS

11.1 Dilute Chemical Warfare Agents

11.1.1 The Monitoring Branch utilizes near drinking water level dilute chemical warfare agents in preparing quality control samples for the IOP. The Monitoring Branch office contains a reference file of material safety data sheets (MSDS) for the dilute CW agents.

11.2 Solvents and Other materials.

11.2.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available which includes, but is not limited to, donning the appropriate laboratory safety attire and working in well-ventilated hoods. The laboratory is responsible for maintaining a current file of Occupational Safety and Health Administrations (OSHA) regulations regarding the safe handling of the chemical specified in this IOP. A reference file of material safety data sheets (MSDS) of materials used in these operations is available at the worksite.

12.0 APPARATUS AND MATERIALS

12.1 Gas chromatograph/Mass Spectrometer (Selective) Detector (MSD)

12.1.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gasses. The capillary column should be directly coupled to MSD.

12.1.2 Column - 30 m x 0.25 mm, ID 1 µm film thickness, silicone-coated fused-silica capillary column (Restek RTX-1701 or equivalent).

12.1.3 The GC should be equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation.

12.2 MSD

12.2.1 MSD - The mass spectrometer detector is one of the powerful used selective detectors in gas chromatography. The MSD uses quadrapoles with an applied electrical current to separate each compound into it's mass/charge particle ratios. This technique allows for the genearation of a spectrum, a "fingerprint", of each compound thus enabling a quick and reliable confirmation.

12.2.2 GC/MSD interface - Any GC-to-MSD interface may be used that gives acceptable calibration points at 2ng or less per injection for each compound of interest and achieves

acceptable tuning performance criteria. For a narrow-bore capillary column, the interface is usually capillary-direct into the mass spectrometer source.

12.3 Data system

12.3.1 Data system - A computer system should be interfaced to the MSD. The system must allow the continuous acquisition and storage on machine-readable media of all data obtained throughout the duration of the chromatographic program.

Appendix I

Required Action Level for GB

Allowable Time Weighted Average (TWA) for GB for 40-hour workweek: 0.0001 mg/cubic meter

Required Action Level:

$$\frac{A\ (mg)}{(m^3)} \times \frac{m^3}{1000\ (l)} \times \frac{1000\ (ug)}{(mg)} \times \frac{B\ (l)}{(min)} \times C\ (min) = D$$

where:
A = TWA, AEL (0.0001 mg/cubic meter)
B = flow Rate
C = sampling Interval
D = required Action Level in µg

For example:

Flow rate in the portable pump: 0.5 l/min
Sampling time: 120 min

Plugging in these values in the formula above:

TWA, AEL for GB: 0.006 ug

Note that there are 1000 ng in 1ug, therefore 0.006 µg = 6.0ng.

Results showing 6.0 ng and higher for GB, under conditions listed above, are to be reported to the Chief, Monitoring Branch and to the POC involved.

The same calculations are used in determining the action levels for HD, VX and GD except that the allowable TWAs are different for these agents.

Appendix II

Surrogate Standard

Surrogate standard - The recommended surrogate is 4-bromofluorobenzene (BFB). Other compounds may be used as surrogates, depending upon the analysis requirements. A stock surrogate solution should be prepared from a stock at a concentration of 0.25ng/ul, in hexane. Each sample undergoing GC/MS analysis must be spiked with 3 µL of the surrogate spiking solution prior to analysis.

Surrogate recoveries - It is necessary that the laboratory evaluate surrogate recovery data from individual samples. The analyst must evaluate the recovery to determine if acceptable system performance was maintained for each analysis. Generally, a 50% or higher recovery is considered acceptable. However, the analyst should base his observation on instrument behavior that day.

Surrogate recovery is calculated as:

Recovery (%) = Concentration (or amount) found/Concentration (or amount) added × 100

If the recovery is not within normal surrogate recovery limits, the following procedures are necessary:

- Check to be sure that there are no errors in the calculations or surrogate solution standard. If errors are found, recalculate the data accordingly.
- Examine chromatograms for interfering peaks and integrated peak areas.
- Check instrument performance. If an instrument performance problem is identified, correct the problem and re-analyze the second DAAMS tube. If no instrument problem is found, the sample should be re-analyzed. If, upon re-analysis, the recovery is again not acceptable, report the data as an "estimated concentration." If the recovery is acceptable in the re-analysis, provide the re-analysis data to the data user.

Appendix III

GC/MSD Tune Evaluation Criteria

1.0 **Tune Verification**

1.1 Prior to the analysis of any samples, including MS/MSDs, blanks, or calibration standards, the analyst must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check solution containing BFB.

2.0 **Frequency of GC/MS Performance Check**

2.1 The instrument performance check solution must be injected once at the beginning of each 12-hour period, during which samples or standards are to be analyzed. The twelve (12) hour time period for GC/MS instrument performance check (BFB), standards calibration (initial or continuing calibration criteria), blank and sample analysis begins at the moment of injection of the BFB analysis that the laboratory submits as documentation of a compliant instrument performance check. The time period ends after twelve (12) hours have elapsed according to the system clock.

3.0 **Procedure for GC/MS Performance Check**

3.1 The analysis of the instrument performance check solution may be performed as an injection of up to 50 ng of BFB into the GC/MS. The instrument performance check solution must be analyzed alone without calibration standards.

4.0 **Technical Acceptance Criteria for GC/MS Performance Check**

4.1 The mass spectrum of BFB must be acquired in the following manner.

4.1.1 Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not background subtract part of the BFB peak.

> *NOTE: All subsequent standards, samples, QPs, QLs, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.*

4.1.2 The analysis of the instrument performance check solution must meet the ion abundance criteria given in Table 3.

5.0 **Corrective Action for GC/MS Performance Check**

5.1 If the technical acceptance criteria are not met, retune the GC/MS system. It may also be necessary to clean the ion source, clean the quadrupole rods, or take other corrective actions to achieve the technical acceptance criteria.

5.2 BFB technical acceptance criteria must be met before any standards, samples, including QPs, or required blanks are analyzed. Any samples or required blanks analyzed when tuning technical acceptance criteria have not been met will require reanalysis.

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Batch: GCMS 2481
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DATA SHEET

MB-Form 11
Revision 9 December 2000

Lab Sample ID:	0304150219-M01	Start: 09:00	End: 10:00		Tube: 05385	Analyst:	SPRiley	04/22/03
Sample Date:	04/15/03 09:00	Flow: 400			GC/MS	GCMS	2481	GCMS10
Sampler:	RDGiles			GB	0 ng	CLEAR	0 mg/m3	<TWA
Org:	Applications Integration Branch E2188			GD	0 ng	CLEAR	0 mg/m3	<TWA
Building:	3938	Room/Item:	135598	HD	0 ng	CLEAR	0 mg/m3	<TWA
Requester:	RHulsey	Phone:	(410) 436-2240	L	0 ng	CLEAR	0 mg/m3	<TWA
Program:	XXX CONFIRMATION							
SOP:								
Batch Position:	2	Status:	WP					
					05385		Reviewed by: SRiley	

Lab Sample ID:	0304150220-M01	Start: 09:00	End: 10:00		Tube: 05383	Analyst:	SPRiley	04/22/03
Sample Date:	04/15/03 09:00	Flow: 400			GC/MS	GCMS	2481	GCMS10
Sampler:	RDGiles			GB	0 ng	CLEAR	0 mg/m3	<TWA
Org:	Applications Integration Branch E2188			GD	0 ng	CLEAR	0 mg/m3	<TWA
Building:	3938	Room/Item:	135599	HD	0 ng	CLEAR	0 mg/m3	<TWA
Requester:	RHulsey	Phone:	(410) 436-2240	L	0 ng	CLEAR	0 mg/m3	<TWA
Program:	XXX CONFIRMATION							
SOP:								
Batch Position:	3	Status:	WP					
					05383		Reviewed by: SRiley	

CW 4/23/03

Appendix IV
Example of Data Sheet showing analytical results

Table 1

Calibration Levels and Typical Concentrations

		GB	GD	HD	L	Surrogate
CAL #	ul (TWA)	Amount(ng)	Amount(ng)	Amount(ng)	Amount(ng)	Amount(ng)
1	1 (0.25)	0.6	0.179	1.004	1.8	0.25
2	2 (0.50)	1.2	0.358	2.008	3.6	0.50
3	3 (0.75)	1.8	0.537	3.012	5.4	0.75
4	5 (1.20)	3.0	0.895	5.020	9.0	1.25
5	7 (1.75)	4.2	1.253	7.028	12.6	1.75

Table 2

Required Action Levels for Air Monitoring Program DAAMS Tubes
(Based on Specified Sampling Parameters)

Agent	Allowable AEL* (mg/m^3)	Sample Volume (liters)	Required Action Level (ng on tube)	Concentration of Agent in the Calibration Standard (ug/ml)
GA, GB, GF	0.0001	24	2.4	0.60
GD	0.00003	24	0.72	0.18
VX	0.00001	24	0.24	0.06
HD, L	0.003	24	72	1.054, 1.808
HN-1, HN-3	0.003	24	72	1.0, 1.0

* Time Weighted Average for 40-hour workweek.

Required Action Level Calculation

$$D(ng) = \frac{A\,(mg)}{m^3} \times \frac{m^3}{1000\,l} \times \frac{1000\,(\mu g)}{(mg)} \times \frac{B\,(l)}{(min)} \times C\,(min) \times \frac{1000\,(ng)}{(\mu g)}$$

Where:
A = AEL
B = flow rate
C = sampling interval
D = required action level

Table 3

BFB Tune Criteria

m/z	Ion Abundance Criteria
50	8.0 – 40.0% of mass 95
75	30.0 – 66.0% of mass 95
95	Base peak, 100% relative adundance
174	50.0 – 120.0% of mass 95
176	93.0 – 101.0% of mass 174

Internal Operating Procedure: Operations Directorate

Monitoring Branch

Title: Operation and Maintenance Procedures for MINICAMS Mounted in a Mobile Vehicle

Division: Chemical Operations

Branch: Monitoring Branch

Building/Area: E3344 and other areas

This internal operating procedure covers operations, methods and procedures of a general nature not covered by a standing operating procedure. This procedure will be effective until rescinded or superseded.

Changes to this procedure will be accomplished by submission of revisions or amendments for approval.

Originator	Jack Mueller	Date
MINICAMS Team Leader:	Jack Mueller	Date
Quality Assurance Coordinator:	Paul L. Stewart	Date
Monitoring Branch Chief	Thomas E. Rosso	Date

Date: December 2003

IOP Number: MT-16

Revision Number: 9

Prepared by: Paul Stewart
X6725

Approved by: T.E. Rosso
X8428

RECORD OF CHANGE

Previous revisions of this document did not incorporate a record of change page.

Revision 6—Incorporated 1ul and 6ul level challenges IAW CASARM Rev#4 draft. Incorporated requirement for separate standards to be used for calibration and Quality Control. Incorporated all other changes required by Revision #4 of the CASARM QA Plan.

Revision 7—Changed 6ul challenge to 4ul challenge throughout the document to conform to the released Revision 4 of CASARM QA Plan.

Revision 8---Added minimum height requirements for GB, GD, HD to section 5.0 and section 6.0 for HD.

Revision 9---Added requirement for all operators to review data at the end of each day and date and initial the hardcopy printout attesting that data was reviewed and is correct. See Sections 5,6,7 & 8.

Added reiteration of requirement to provide data to MINICAMS group for review on a monthly basis even if this requires that the data be mailed/shipped to the MINICAMS team for review. See sections 3, 5,6,7,&8.

Added clarification of challenge/clearance requirements to Section 11. Clarified Requirement to challenge before and after Headspace monitoring to clear samples and how to input data into appropriate MBFORM 37 to facilitate data entry into LIMS.

Table of Contents

Operator's Statement: I have read, or have had read to me the procedures in this IOP. I, by my signature below, indicate that I thoroughly understand and agree to abide by these instructions.

Signature	Date	Signature	Date

Supervisor's Statement: I have personally reviewed this IOP and, to the best of my knowledge, believe that the information listed herein is correct.

Supervisor: _____ Date: _____

OPERATION AND MAINTENANCE PROCEDURES
FOR MINICAMS MOUNTED IN A MOBILE CONFIGURATION

1.0 Purpose

The purpose of this IOP is to describe the operation and maintenance of the MINICAMS Agent Monitoring System mounted in a Mobile Configuration.

2.0 MINICAMS Monitoring Concept.

The MINICAMS system is currently installed in a specially designed MAP or modified pickup truck and it used for near real time monitoring at various sites throughout the Continental United States and other OCONUS areas as required.

The MINICAMS provides workers involved in remediation operations with near-real time monitoring for the presence of airborne chemical warfare material in the workplace. **Each system is calibrated and challenged at the beginning of each operational day. Continuous monitoring is achieved through the use of a heated sampling line.** The sample is drawn through the line into the MINICAMS unit, which desorbs and analyzes the sample during the two-minute purge time. The data, collected from each MINICAMS, are stored on a floppy disk and/or on a hard copy printout. This serves as a historical record of (no) agent release.

2.1 Definition of the Presence of Chemical Agent.

During mobile operations, chemical agent is defined as present if detected by the MINICAMS in a concentration greater than or equal to the Time Weighted Average* (TWA) level for three consecutive cycles.

A member of the monitoring team monitors the performance of the MINICAMS on a daily basis. The MINICAMS is challenged each workday using certified standards for the agent(s) under test to assure that the MINICAMS is operating correctly

2.2 Calibration Standards and QC Standards

Separate standards are required for calibration and QC check of the MINICAMS. The MINICAMS will be calibrated for the agent(s) of interest with a 4ul injection of the Calibration standard. To check the calibration curve, two QC challenges will be performed, the first challenge is a 1ul injection of the agent(s) of interest and the second check is a 4ul injection of the agent(s) of interest. The 1ul injection is acceptable if the analytical result is within 50% of the true concentration. The 4ul injection is acceptable if the analytical result is within 25% of the true concentration.

NOTE: The operator may make a 2nd calibration injection that will be averaged with the first calibration injection by the MINICAMS software if the following procedure is followed: e.g. perform a calibration injection, then press enter before performing the 2nd injection, then press enter again to average the two injections.

A 4ul QP injection as used in this document, is equal to "1 TWA"
Challenge in the end of the sample line=The end located away from the injection port on the MINICAMS.
* The terms Time Weighted Average and Airborne Exposure Limit (AEL) can be used interchangeably. TWA is used in this document to be consistent with the MINICAMS's software.

NOTE: Calibration Standards and QC Standards are made up in both 5'minute and 10'minute concentrations. Either the 5 minute or 10 minute standard may be used to calibrate and challenge the MINICAMS depending on how the MINICAMS is set up for agent monitoring. e.g. 5'minute standards are generally used for personnel safety monitoring to provide near real time information.

3.0 Control Samples.

A MINICAMS sample is defined as the volume of air that is sampled during one automatic cycle of the instrument. All challenges of the dilute chemical agent are injected into the end of the heated sampling line during the sampling period of the MINICAMS cycle. MINICAMS in the VX mode will have injections made onto the fluoride conversion pad. MINICAMS in the L mode will have injections made into the sample probe. MINICAMS equipped with a loop-sampling module will be calibrated and challenged IAW the specific calibration procedures specified later in this IOP.

A record of the test date, time, heated sample line serial number, MINICAMS result and the operator's initials will be maintained for sample line challenges on MBFORM-4. See sample attached to this IOP. **Flow rate checks will be recorded in the Comment section of the form OR if a flow meter is in line, the operator will initial the flow rate of the "params" printout.** This record will be maintained with the MINICAMS during operations. The data are transported back to the Monitoring Branch at the end of each operational month for final documentation review and storage.

Note: The following will apply only when performing personnel safety monitoring.
End of day challenges that fail to meet the acceptance criteria will not require an additional challenge or re-calibration of the MINICAMS if monitoring is completed for the operational day.

All QC challenges of chemical agent monitors will be initialed on the hard copy printout of the operating parameters after each calibration and challenge. All QC challenges during operations that require more than one daily agent challenge will be printed out on hard copy. The data are also stored on the hard drive of the MINI LINK computer and are archived for subsequent storage.

NOTE: THE MONTHLY MINICAMS DATA WILL BE SHIPPED BY FEDEX OR EQUIVALENT, TO THE ECBC SITE FROM EACH OFFSITE LOCATION FOR REVIEW AND RETENTION AT THE END OF EACH MONTH.

4.0 Preventive Maintenance Procedures.

4.01 CHECK THAT THE MINICAMS PM STICKER IS CURRENT.

4.02 Verify that the unit is in the correct agent and concentration mode.

4.03 Verify that the unit is in the run (not calibrate) mode.

4.04 Verify that the recorder power switch is on (if applicable).

4.05 Verify that the alarm lamp, and horn come on when the MINICAMS is challenged.

4.06 Verify that no error message appears on the display.

4.07 Verify that each compressed-gas cylinder contains at least 200 psig. If not, change the cylinder. If monitoring for L, verify that the EDT is at 30-35 psi.

4.08 Check the amount of printer paper remaining. Replenish, if necessary.

4.09 Heated sample lines used for Lewisite (L) monitoring, require "**Back Flushing**" with ~**200mls of acetone** followed by ~**200 mls of 2proponal** to eliminate L carryover after each week of operation, or as required to keep the instrument in control.

4.10 Replace the Pre-Concentrator Tube (PCT) and reactor tube (if applicable) weekly or as needed. i.e. Response is getting lower or area getting broader widths.

4.11 Check the external gas lines and fittings for leaks.

NOTE: Manufacturers suggested flow rates:

XSD-400 $\pm$ 50 MILL/MINUTE
FPD-600-1000 MILLS/MINUTE
LOOP-100 $\pm$ 15 MILLS/MINUTE

5.0 CALIBRATION PROCEDURE FOR VX, GB, GD and HD USING THE FPD

5.01 At the beginning of each workweek, get a new calibration standard and a new QC standard from the laboratory or from the MAP if operating in the field.

5.02 These standards should be kept cold **except** when in use, to minimize deterioration of the standards due to evaporation.

5.03 Unused standards shall be destroyed when no longer required.

5.04 Check that the MINICAMS PM sticker is current.

5.05 Change the PCT weekly as a minimum. Operator shall document that weekly replacements has been made in comments section of MBFORM-4.

5.06 If the MINICAMS is programmed for VX operation, replace V-G pad at the end of the sample line situated away from the point of attachment to the MINICAMS at least weekly. V-G pad may require changing more frequently during rainy or very humid weather.

5.07 Check MINICAMS operational parameters for errors. If there are no errors, Inject a 4ul " challenge" of 10 minute COMBO QC standard and 4 ul of the 10 minute VX QC standard onto the V-G filter pad at the end of the heated sample line when the MINICAMS display screen reads INJECT. Label this shot as "CHALL" on the chromatogram. i.e. "4ul CHALL JAM"

5.08 Press the PARAM button until RUN is flashing in the lower left corner, then press the INCR button until CAL is flashing and press ENTER. When the word INJECT appears on the display screen, perform two injections, 4ul of the 10 minute COMBO CALIBRATION standard and 4ul of the 10 minute VX CALIBRATION standard on the surface of the V-G filter pad located at the end of the heated sample line.

5.09 During the Purge cycle, press F6 to label this chromatogram as a calibration and enter the operator's initials and challenge amount on the chromatogram. (i.e. 4ul CAL JAM).

NOTE: **The minimum acceptable VX height must be 100 nanoamps** before the injection can be calibrated. If VX height cannot achieve acceptable limits, the Photomultiplier voltage should be increased by increments of 25 volts until VX height is acceptable. If Photomultiplier voltage is already set to the maximum setpoint of 1250 volts and the minimum height for VX still cannot be achieved, the instrument should be removed from service for repairs and replaced with a working MINICAMS. **Minimum acceptable height for: HD=500, GB=1000, GD=300.**

5.10 After the printer completes the print out, write the **CAL HEIGHT in the CAL/H** column on the individual QC data sheet (**MBFORM 4**). Press ENTER from the main screen. (This action will normalize the found concentrations to 1.00 on the MINICAMS screen and the printer will print as calibration.)

5.11 Press the PARAM button on the MINICAMS until CAL is flashing on the lower left of the screen, press INCR button until RUN is flashing and then press ENTER.

5.12 Two QC challenges shall be used to check calibration. **First**, Perform a 1ul injection of the 10 minute COMBO QC standard and a 1ul injection of the 10 minute VX QC standard onto the V-G pad at the end of the heated sample line when the screen reads INJECT. Result shall be within 50% of true concentration.
During the Purge cycle, press F6 to label this chromatogram as a challenge and enter the operator's initials on the chromatogram. i.e. "1ul CHALL JAM"

Second, perform a 4ul injection of the 10 minute COMBO QC standard and a 4ul injection of 10 minute VX QC Standard onto the V-G pad at the end of the heated sample line, when the screen reads INJECT. Result shall be within 25% of true concentration.
During the Purge cycle, press F6 to label this chromatogram as a challenge and enter the operator's initials on the chromatogram. i.e. "4ul CHALL JAM"

5.13 An audible alarm will sound if a concentration greater than 0.70 TWA is detected for any of the selected agents. Press the ENTER button on the MINICAMS to silence this alarm horn. (The alarm light will continue to flash until the concentration falls below 0.70 TWA.)

5.14 Press the F4 key and print out the chromatogram screen. When the screen reads inject, press the Print key on the MINICAMS to print out the parameters. **This will provide the instrument flow rate**, which the operator will date and initial to indicate that flow rate is correct.

5.15 Record each found concentration on the QC data sheet under the FC 1 column. Each of the concentrations must be acceptable. . If not, deselect any agents, which passed the first challenge, and repeat steps 5.08 through 5.15 above and record data under the FC2 columns.

NOTE: To deselect an agent, press the PAGE button 4 times until you are on the agent selection screen. Press the PARAM button until the asterisk is blinking beside the agent to be deselected. Press the ENTER button and the asterisk will disappear. At this time, this agent has been deselected.

5.16 If the second challenge is still not within the acceptable limits, perform required corrective actions and perform another calibration. Repeat steps 5.08 through 5.15 again and record found concentration under the FC3 column and 2nd CAL HT under corrective action (CA) column on MBFORM-4.

NOTE: **If the third challenge is still out of range, this instrument must either be removed from service or additional corrective action must be performed and documented on the QC data sheet.** After the corrective action has been taken, the instrument must be re-calibrated and the data recorded on the next line. If the instrument is replaced, a new QC data sheet must be started and calibrations and challenges must be performed as indicated above.

5.17 After acceptable challenges have been performed, reselect any deselected agents by paging to the agent selection screen (See note under paragraph 5.15), press PARAM until the asterisk blinks beside the agent to reselect and press the ENTER button and the asterisk will reappear and the agent has been reselected.

5.18 Once all agents have been reselected, page back to the main screen and press PRINT to print out operating parameters.

5.19 Operator shall initial the parameters printout next to the flow rates and attach the paperhanger to the top of the printer paper.

5.20 Make sure the operator's initials, CAL Height, Heated sample line serial number and challenge data are recorded properly on the QC data sheet.

5.21 Plot the 1ul and 4ul QC challenge shots on the QC performance chart located on MBFORM-4. Both the 1ul and the 4ul QC challenge shots shall be within the acceptance limits listed on the performance chart on MBFORM-4 to be acceptable.

NOTE: AT THE END OF EACH WORKDAY, EACH OPERATOR ON SITE SHALL REVIEW ALL MINICAMS PRINTOUT DATA (HARDCOPY) AND CORRESPONDING QP DATA SHEET ENTRIES FOR CONFORMANCE TO REQUIREMENTS. EACH OPERATOR SHALL REVIEW THE DATA FROM EACH MINICAMS, (EVEN IF THAT OPERATOR DID NOT OPERATE THAT MINICAMS), THEN DATE AND INITIAL THE HARDCOPY PRINTOUT AT THE END OF THE DAILY PRINTOUT ATTESTING THAT THE DATA HAS BEEN REVIEWED AND IS CORRECT. i.e. data reviewed by JAM 1/6/04, data reviewed by JRF 1/6/04, data reviewed by MPP 1/6/04. This will provide an additional check that the data is correct and accurate on a daily basis.

NOTE: The MINICAMS operator shall start a new QC data sheet, MBFORM-4 when the MINICAMS is changed and/or on the first operational day of each month. The data (QC data sheets) for the previous month shall be collected and **turned in or SHIPPED** to the MINICAMS group for review and filing. **See note in section 3 for shipping information.**

6.0 CALIBRATION PROCEDURE FOR HD USING THE FPD

6.01 At the beginning of each workweek, get a new calibration standard and a new QC standard from the laboratory or from the MAP if operating in the field.

6.02 These standards should be kept cold except when in use, to minimize deterioration of the standards due to evaporation.

6.03 Unused standards shall be destroyed when no longer required.

6.04 Check that the MINICAMS PM sticker is current.

6.05 Change the PCT weekly as a minimum. Operator shall document that weekly replacements has been made in comments section of MBFORM-4.

6.06 If there are no errors, Inject a 4ul "challenge" of 10 minute COMBO QC standard or 10'minute HD QC standard at the end of the heated sample line when the MINICAMS display screen reads INJECT. Label this shot as "4ul CHALL" on the chromatogram. i.e. "4ul CHALL JAM"

6.07 Press the PARAM button until RUN is flashing in the lower left corner, then press the INCR button until CAL is flashing and press ENTER. When the word INJECT appears on the display screen, perform a 4ul injection of the 10'minute COMBO calibration or 10'minute HD calibration standard into the end of the heated sample line.

6.08 During the Purge cycle, press F6 to label this chromatogram as a calibration and enter the operator's initials and challenge amount on the chromatogram. (i.e. 4ul CAL JAM).

6.09 After the printer completes the print out, write the **CAL HEIGHT in the CAL/H** column on the individual QC data sheet (**MBFORM 4**). Press ENTER from the main screen. (This action will normalize the found concentrations to 1.00 on the MINICAMS screen and the printer will print as calibration.) **Minimum acceptable height for HD=500.**

6.10 Press the PARAM button on the MINICAMS until CAL is flashing on the lower left of the screen, press INCR button until RUN is flashing and then press ENTER.

6.11 Two challenges shall be used to check calibration. **First**, perform a 1ul injection of the 10'minute COMBO QC standard or a 1ul injection of the 10'minute HD QC standard, when the screen reads INJECT. Result shall be within 50% of true concentration.
During the Purge cycle, press F6 to label this chromatogram as a challenge and enter the operator's initials on the chromatogram. i.e. "1ul CHALL JAM"

Second, perform a 4ul injection of the 10'minute COMBO QC standard or a 4ul injection of 10'minute HD QC Standard, when the screen reads INJECT. Result shall be within 25% of true concentration.
During the Purge cycle, press F6 to label this chromatogram as a challenge and enter the operator's initials on the chromatogram. i.e. "4ul CHALL JAM"

6.12 An audible alarm will sound if a concentration greater than 0.70 TWA is detected for any of the selected agents. Press the ENTER button on the MINICAMS to silence this alarm horn. (The alarm light will continue to flash until the concentration falls below 0.70 TWA.)

6.13 Press the F4 key and print out the chromatogram screen. When the screen reads inject, press the Print key on the MINICAMS to print out the parameters. This will provide the instrument flow rate, which should be dated and initialed by the operator on the chromatogram.

6.14 Record each found concentration on the QC data sheet under the FC 1 column. Each of the concentrations must be acceptable. If not, repeat steps 6.07 through 6.14 above for agents requiring a 2nd challenge and record data under the FC2 column. Make sure that the minimum CAL Height is maintained.

6.15 If the second challenge is still not within the acceptable limits, perform any required corrective actions and perform another calibration. Repeat steps 6.07 through 6.14 again and record found concentration under the FC3 column and 2nd CAL HT under CA column.

NOTE: **If the third challenge is still out of range, this instrument must either be removed from service or additional corrective action must be performed and documented on the QC data sheet.** After the corrective action has been taken, the instrument must be re-calibrated and the data recorded on the next line(s). If the instrument is replaced, a new QC data sheet must be started and calibrations and challenges must be performed in accordance with above requirements.

6.16 Page back to the main screen and press PRINT to print out operating parameters.

6.17 Operator shall initial the parameters printout next to the flow rates and attach the paperhanger to the top of the printer paper.

6.18 Make sure the operator's initials, CAL Height, Heated sample line serial number and challenge data are recorded properly on the QC data sheet.

6.19 Plot both the 1ul and the 4ul QC challenge shots on the QC performance charts located on MBFORM-4. Both the 1ul and the 4ul QC challenge shots shall be within the acceptance limits listed on the performance chart on MBFORM-4 to be acceptable.

NOTE: AT THE END OF EACH WORKDAY, EACH OPERATOR ON SITE SHALL REVIEW ALL MINICAMS PRINTOUT DATA (HARDCOPY) AND CORRESPONDING QP DATA SHEET ENTRIES FOR CONFORMANCE TO REQUIREMENTS. EACH OPERATOR SHALL REVIEW THE DATA FROM EACH MINICAMS, (EVEN IF THAT OPERATOR DID NOT OPERATE THAT MINICAMS), THEN DATE AND INITIAL THE HARDCOPY PRINTOUT AT THE END OF THE DAILY PRINTOUT ATTESTING THAT THE DATA HAS BEEN REVIEWED AND IS CORRECT. i.e. data reviewed by JAM 1/6/04, data reviewed by JRF 1/6/04, data reviewed by MPP 1/6/04. This will provide an additional check that the data is correct and accurate on a daily basis.

NOTE: The MINICAMS operator shall start a new QC data sheet, MBFORM-4 when the MINICAMS is changed and/or on the first operational day of each month. The data (QC data sheets) for the previous month shall be collected and **turned in or SHIPPED** to the MINICAMS group for review and filing. See note in section 3 for shipping information.

7.0 CALIBRATION PROCEDURE FOR HD/XL USING THE XSD MINICAMS

7.01 At the beginning of each workweek, get a new calibration standard and a new QC standard from the laboratory or MAP if operating in the field.

7.02 These standards should be kept cold except when in use, to minimize deterioration of the standards due to evaporation.

7.03 Unused standards shall be destroyed when no longer required.

7.04 Check that the MINICAMS PM sticker is current.

7.05 Change the pre-concentrator tube and the reactor tube each Monday or first day of the workweek or after cleaning heated sample lines. Operator shall document that weekly replacements has been made in comments section of MBFORM-4.

7.06 Check MINICAMS operational parameters for errors. If there are no errors, Withdraw 4ul of 1.79 ng/ul from the lewisite QC standard into a syringe and 4ul of 10'minute HD QC standard into another syringe and inject both into the probe end of the sample line when the light on the probe controller box illuminates. This occurs at time 245 on the MINICAMS screen. Label this shot as "CHALL" on the chromatogram. i.e. "4ul CHALL JAM".

7.07 From the main screen, press the PARAM button until RUN is flashing in the lower left corner, then press the INCR button until CAL is flashing and press ENTER.

7.08 Withdraw 4ul of 1.79 ng/ul from the lewisite calibration standard into a syringe and 4ul of 10 minute HD calibration standard into another syringe and inject both into the probe end of the sample line when the light on the probe controller box illuminates. This occurs at time 245 on the MINICAMS screen. Label this shot as "CAL" on the chromatogram. i.e. "4ul CAL JAM".

7.09 Allow the printer to print and write the **CAL HEIGHT in the CAL/H** column on the individual QC data sheet (**MBFORM 4**). Press ENTER from the main screen. (This action will normalize the found concentrations to 1.00 on the MINICAMS screen and the printer will print as calibration.)

7.10 Press F6 to label this chromatogram as a calibration and enter the operator's initials and challenge amount. (i.e. 4ul CAL JAM).

The minimum acceptable HD calibration height should be 250 nanoamps or greater. The minimum acceptable L calibration height must 100 nanoamps. If the heights are slightly lower, (i.e.200 & 85) but the QP's are within acceptable range, operations may continue at the operators discretion. Corrective action is required before operations can continue the next day, if the above conditions can't be met.

NOTE: The need for corrective action should be considered once the calibration height falls to 300 nanoamps for HD and 125 nanoamps for Lewisite to preclude out-of-control operations

7.11 Press the PARAM button on the MINICAMS until CAL is flashing on the lower left of the screen, press INCR button until RUN is flashing and then press ENTER.

7.12 Two challenges shall be used to check calibration.
First, perform a 1ul injection of the Lewisite QC standard and a 1ul injection of the 10 minute HD QC standard as follows: withdraw 1ul of 1.79 ng/ul from the lewisite QC standard into a syringe and 1ul of 10 minute HD QC standard into another syringe and inject both into the probe end of the sample line when the light on the probe controller box illuminates. This occurs at time 245 on the MINICAMS screen.
During the purge cycle, Press F6 to label this chromatogram as a challenge. Enter the operator's initials. i.e. "1ul CHALL JAM". Result shall be within 50% of true concentration.

Second, perform a 4ul injection of the Lewisite QC standard and a 4ul injection of the 10 minute HD QC standard as follows: withdraw 4ul of 1.79 ng/ul from the lewisite QC standard into a syringe and 4ul of 10 minute HD QC standard into another syringe and inject both into the probe end of the sample line when the light on the probe controller box illuminates. This occurs at time 245 on the MINICAMS screen.

7.13 During the purge cycle, Press F6 to label this chromatogram as a challenge. Enter the operator's initials. i.e. "4ul CHALL JAM". Result shall be within 25% of true concentration.

7.14 When the challenge concentration(s) is printed out, an audible alarm will sound if any of the concentrations are greater than the alarm set point. Press the ENTER button to silence this alarm horn. (The alarm light will continue to flash until all of the concentrations fall below the alarm set point.) If monitoring for HD only, set the alarm set point level at .70 TWA.

NOTE: If monitoring for Lewisite, set the alarm set point level at 0.25.

7.15 Press the F4 key and print out the chromatograms. After the word Inject appears on the screen, press the Print button on the MINICAMS and print Parameters.

7.16 Record the found concentration(s) on the QC data sheet under the FC1 column. **The 1ul concentration must be within 50% of true concentration for HD and Lewisite. The 4ul concentration must be within 25% of true concentration for HD and Lewisite.**

7.17 If one of the agents is not within the specified limit, deselect the passing agent by pressing the PAGE button 4 times until you are on the agent selection page. Press the PARAM button until the asterisk blinks beside the agent to be deselected and press ENTER. Repeat steps 7.07 through 7.16 above and record data under the FC2 column.

7.18 If the second challenge is still not within the acceptable limits, perform and document corrective action, repeat steps 7.07 through 7.16 again and record FC under F3 column & CAL HT under CA column.

NOTE: **If a third challenge is still out of range, this instrument must either be removed from service or additional corrective action must be performed and documented on the QC data sheet.** At that time re-calibration can be performed and the data recorded on the next line. If the instrument is replaced, a new QC data sheet must be started and calibrations and challenges must be performed as indicated above.

7.19 After all challenges are acceptable, reselect all agents by again paging to the agent selection screen (see 7.17) and press the PARAM button until the asterisk is flashing next to the agent to be reselected and press ENTER. The asterisk will be replaced with XSD. Press ENTER again and the asterisk will reappear and the agent has been reselected. Page back to the main screen.

7.20 Press the F4 key and print out the chromatograms. After the word Inject appears on the screen, press the Print button on the MINICAMS and print Parameters.
Initial the printout and attach the paperhanger to the top of the printer paper.

7.21 Make sure the operator initials, heated sample line serial number, calibration and challenge data are recorded properly on the QC data sheet.

7.22 Archive MINILINK data every Friday (or last operational day of the week) in accordance with the archive procedure. Note: Monday to Monday is also acceptable as long as the 1000 data storage isn't exceeded.

7.23 Plot both the 1ul and the 4ul QC challenge shots on the QC performance charts located on MBFORM-4. Both the 1ul and the 4ul QC challenge shots shall be within the acceptance limits listed on the performance chart on MBFORM-4 to be acceptable.

7.24 Mobile MINICAMS shall be challenged with a LLC within five hours (if operations are expected to exceed five hours) and a LLC at the end of the day. Acceptance criteria is $\pm$ 50% of TC.

NOTE: Heated sample line shall be **"Back Flushed"** with ~200mls of acetone followed by ~200mls of 2PROPANOL every week, or as necessary, to eliminate the L carryover. (carryover greater than 0.2 for 1 cycle).

Note: When monitoring for Lewisite, a bypass line should be used to keep the line filled with fresh air and evacuate the line of EDT.

NOTE: AT THE END OF EACH WORKDAY, EACH OPERATOR ON SITE SHALL REVIEW ALL MINICAMS PRINTOUT DATA (HARDCOPY) AND CORRESPONDING QP DATA SHEET ENTRIES FOR CONFORMANCE TO REQUIREMENTS. EACH OPERATOR SHALL REVIEW THE DATA FROM EACH MINICAMS, (EVEN IF

THAT OPERATOR DID NOT OPERATE THAT MINICAMS), THEN DATE AND INITIAL THE HARDCOPY PRINTOUT AT THE END OF THE DAILY PRINTOUT ATTESTING THAT THE DATA HAS BEEN REVIEWED AND IS CORRECT. i.e. data reviewed by JAM 1/6/04, data reviewed by JRF 1/6/04, data reviewed by MPP 1/6/04. This will provide an additional check that the data is correct and accurate on a daily basis.

NOTE: The MINICAMS operator shall start a new QC data sheet, MBFORM-4, when changing MINICAMS and/or on the first day of each month. The data (QC data sheets) for the previous month shall be collected and turned in OR SHIPPED to the MINICAMS group for review and filing. See note in section 3 for shipping information.

8.0 CALIBRATION PROCEDURE FOR PS, CG, CK, AND CF WHEN USING MINICAMS EQUIPPED WITH A LOOP SAMPLING MODULE. NOTE: SEE APPENDIX 1 FOR GAS STANDARD BAG FILLING PROCEDURES.

8.01 Get new Gas Standard Bags from the Laboratory (or MAP if operating in the field) at the beginning of each workday.

Note: If monitoring for Phosgene (CG), also get a high concentration gas standard bag and attach to the heated sample line end and allow to run for 4 cycles until equilibration occurs.

8.02 Check MINICAMS (equipped with a loop sampling GC module) for errors, if none, attach the gas sample bag containing a 1 TWA concentration of the agent of interest to the INLET (throat/Injection port of the MINICAMS) while in purge and open bag until 1 complete thread is showing on the valve, after 1st report, press F6 key and label chromatogram with 1 TWA and Initials. Allow the printer to print. .i.e. "1 TWA CHALL JAM"

NOTE: Paragraph 8.03 below will be done "every other week" during routine maintenance to check the propagation of CG through the heated sample line.

8.03 Check MINICAMS (equipped with a loop sampling GC module) for errors, if none, attach the gas sample bag containing a 1 TWA concentration of the agent(s) of interest, to the end of the heated sample line, **(the end opposite from end attached to the MINICAMS inlet)** and open bag. Allow the printer to print.

8.04 From the main screen, press the PARAM button until RUN is flashing in the lower left corner, press the INCR button until CAL is flashing and then press enter.

8.05 Allow the printer to print again (approximately 120 seconds) and write **CAL HEIGHT** on the individual QC data sheet under the **CAL/H column**. Press ENTER from the main screen. (This action will normalize the found concentrations to 1.00 on the MINICAMS screen and the printer will print as calibration.)

8.06 Press the F6 key on the MINILINK computer and label the chromatogram as a calibration and enter the operator's initials. i.e. "1TWA CAL JAM"

8.07 Press the PARAM button until CAL is flashing on the lower left hand side of the screen, press the INCR button until Run is flashing and press ENTER.

8.08 After final QP reports and prints, press F6 key and label chromatogram as before. . i.e. "1TWA CHALL JAM" If found concentrations are acceptable record found concentrations under the FC1 column. If 1st challenge fails, operator shall perform a 2nd challenge and record results under FC2 and label chromatogram as before. i.e. "1 TWA CHALL JAM"

8.09 If the 2nd challenge found concentration(s) are not within the acceptable range, (0.75-1.25) record the found concentration(s) under FC2 column. place the MINICAMS into the CAL mode and perform another calibration challenge. After result is printed, Press ENTER from the main screen. (This action will normalize the found concentrations to 1.00 TWA on the MINICAMS screen and the printer will print as calibration.

8.10 Use the F6 key to label the chromatogram as a calibration and enter the operator's initials and concentration amount as before. . i.e. "1TWA CAL JAM"

8.11 Press the PARAM button until CAL is flashing on the lower left hand side of the screen, press the INCR button until RUN is flashing and press enter.

8.12 After final QP reports and prints, press the F6 key and label the chromatogram as before. i.e. "1TWA CHALL JAM". If the found concentrations are acceptable, record found concentration(s) under FC3 column and CAL/HT under CA column.

8.13 Press the F4 key on the MINILINK to print the chromatogram.

NOTE: **If a third challenge is still out of range, this instrument must either be removed from service or additional corrective action must be performed and documented on the QC data sheet.** At that time recalibration can be performed and the data recorded on the next line. If the instrument is replaced, a new QC data sheet must be started and calibrations and challenges must be performed.

8.14 Initial the printout and attach the paperhanger to the top of the printer paper.

8.15 Archive MINILINK data every Friday (or last operational day of the week) in accordance with the archive procedure. **Note: Monday to Monday is also acceptable as long as the 1000 data storage isn't exceeded.**

NOTE: AT THE END OF EACH WORKDAY, EACH OPERATOR ON SITE SHALL REVIEW ALL MINICAMS PRINTOUT DATA (HARDCOPY) AND CORRESPONDING QP DATA SHEET ENTRIES FOR CONFORMANCE TO REQUIREMENTS. EACH OPERATOR SHALL REVIEW THE DATA FROM EACH MINICAMS, (EVEN IF THAT OPERATOR DID NOT OPERATE THAT MINICAMS), THEN DATE AND INITIAL THE HARDCOPY PRINTOUT AT THE END OF THE DAILY PRINTOUT ATTESTING THAT THE DATA HAS BEEN REVIEWED AND IS CORRECT. i.e. data

reviewed by JAM 1/6/04, data reviewed by JRF 1/6/04, data reviewed by MPP 1/6/04. This will provide an additional check that the data is correct and accurate on a daily basis.

NOTE: The MINICAMS operator shall start a new QC data sheet, MBFORM-4 when a new MINICAMS is installed and/or on the first day of every month. The data (QC data sheets) for the previous month shall be collected and turned in or SHIPPED **to the MINICAMS group for review and filing. See note in section 3 for shipping information.**

9.0 ARCHIVE PROCEDURE FOR MINILINK SYSTEM. NOTE: USE **FORMATTED** 3.5 in DISKETTES.

NOTE: This procedure augments the CMS Operators Manual and may be used with MINILINK system.

MINILINK IS SET ON THE VISUAL STRIP CHART SCREEN DURING NORMAL OPERATIONS.

ARCHIVE DATA WHEN ARCHIVE IS FLASHING IN THE UPPER RIGHT CORNER OF COMPUTER SCREEN.

When ARCHIVE is flashing in the upper right corner of the computer screen, the hard drive is approaching 1000 stored chromatograms. If the data is not archived, the hard drive becomes full and the MINILINK automatically starts overwriting previously stored data. Once overwritten the data is impossible to retrieve.

TO ARCHIVE DATA:

1. Go to main menu and select #7 Archive Data.

2. This screen has prompts to remind the operator to use formatted disks, date the disks with proper date (date of data to be archived, which has changed with the new 1999 y2k software. Now, enter the date that the archive procedure is performed. The new software will archive back to the last date archived.

3. Label the disk with the location/position, start and end date, and disk number. E.g. BRA-1, Data from 8/19/98 to 8/15/98, Disk #1. NOTE: It usually requires 4-6 disks to complete archive procedure.

4. Press ESCAPE on upper left corner of keyboard. At C:\ MINILINK > prompt, type ARC (space) 081998 per example above, and press ENTER

5. Type y and press ENTER to archive data. The bottom line on the screen will read "Do you want to delete files at end of archive procedure?" Type y unless you want more than 1 copy of the data. The data will then be deleted from the hard drive at the end of the archive procedure to make room for additional data and the flashing ARCHIVE will be eliminated from the strip chart screen.

6. The operator will be prompted to insert a disk into the computer and press any key to begin the archive procedure.

7. As each disk is filled, the operator will be prompted to insert disk #2, #3 etc. and press any key to start. Repeat this procedure until all data is copied to the diskettes. The program prompts the operator for each operation.

8. When all files are copied, the program will read "All copies filed" and the C:\MINILINK> prompt will reappear. Type MLINK to return to the main menu.

9. Document that the data has been successfully archived by writing "Data arced" in the comment section of the QC Data sheet and store all disks until the disk holder is full.

10. Replace full diskette container with a new container and return the old data filled container to the data storage area.

11. Each container will be labeled with the position name and number and given to the SciTech production department for downloading all disk data onto a Zip Drive and then cleaning diskettes for reuse.

NOTE: For additional information on archiving data, refer to the CMS MINILINK Operators Manual.

IF PROBLEMS OCCUR, REFER TO THE FOLLOWING:

If a problem occurs during the archiving process, a message will appear with choices for the operator, e.g. "Reading on drive B—Abort, Retry, Ignore etc. Press "R" for retry. If message still appears, remove disk and add the letter "A" behind the disk number and insert a new disk and press enter. If the message appears again, Press "I" each time the message appears until the program gets past the bad sector. All data will be stored on the disk. Be sure and label the disk with "B" behind the disk number. Example: BRA-1, Data from 8/19/98 to 8/15/98, Disk 1A, Then the replacement disk will have the same data except the label will be Disk 1B.

10.0 DAILY STARTUP/SHUTDOWN AND MONITORING OPERATION REQUIREMENTS

10.1 Check the Oil in the Generators and Vehicle before starting operations.

10.2 Check vehicle and generators to make sure all tanks are full of gas.

10.3 Perform all requirements on checklist MBFORM-35 before starting vehicles or Generators. Retain completed checklists with vehicle.

10.4 While MINICAMS are still connected to shore power, start up the Generator and allow to run approximately 5 min.

10.5 Turn off the power strip inside the truck and unplug this cord from shore power. Plug the power cord into the 4 outlet gang box, which is connected to the generator. Turn on the power strip inside the truck and verify there are no errors after warm-up.

10.6 Plug in the heated sample line cord(s) to the gang box and allow 10-15 min. for heated sample lines to get hot.

10.7 During this time, get the agent standard(s) and sign paperwork in the MAP.

10.8 Perform the AM calibration and challenge and record the found concentrations and the actual flowrate from the printout on MBFORM-4.

10.9 Proceed to operational site and contact the site safety supervisor to find out where to set-up the MINICAMS truck.

10.10 Set the heated sampling line in position for monitoring and perform an onsite LLC and record challenge data on MBFORM-4. Be sure to label each chromatogram with the site information. Once the onsite challenge passes, notify the site supervisor that the MINICAMS' are in control.

NOTE: Any time the truck moves to a different location or after 4 to 5hrs of monitoring, re-challenge the MINICAMS by performing a low level QP challenge for all agents of interest using the QC standard. Injections shall be made into the end of the heated sample line.

All MINICAMS challenges must be acceptable prior to continuing monitoring operations. i.e. low level challenge must be within ± 50% of true concentration and high level challenge must be within ±25% of true concentration. If a challenge fails to meet requirements, a 2nd challenge is permitted. If the 2nd challenge fails, re-calibration is required.

End of day challenges that fail to meet the acceptance criteria will not require additional challenges or re-calibration of the MINICAMS if monitoring is completed for the operational day.

Note: at the end of each operational day, top off all generator gas tanks and take metal cans and refill in the am before work.

11.0 HEADSPACE MONITORING FOR ENVIRONMENTAL SAMPLES

The MINICAMS must be calibrated and challenged successfully IAW with sections 5,6, 7 or 8 above as appropriate, within 30 minutes of starting HEADSPACE MONITORING or

SCRAP PPE MONITORING, OR have been subjected to a successful 1ul QP challenge prior to proceeding with paragraph 11 below.

To Clear samples, the MINICAMS must be successfully challenged with a 1ul QP with the agent(s) of interest after completion (within 30 minutes) of headspace environmental samples monitoring.

NOTE REQUIREMENT: To Clear samples: See note at 11.1.10 below.
Successful 1ul QP challenge before monitoring.
Successful 1ul QP challenge after monitoring.

The MINICAMS operator will fill out a MBFORM-37a,b or c as applicable, for each MINICAMS used to clear samples, to provide information for entering the clearance information into the LIMS. A separate MBFORM-37 must be filled out for each MINICAMS used to clear samples.

CAUTION NOTE: Do not put sample clearance information from 2 MINICAMS on the same MBFORM-37.
Normally, soil sample headspace analysis will be conducted using the MINICAMS. If DAAMS tubes are used in place of MINICAMS, start the following sequence at step 11.1.11. DAAMS tubes must be used to confirm all positive MINICAMS readings. Headspace Monitoring Procedures of soil samples using the MINICAMS and DAAMS are as follows:

11.1 Soil Headspace Monitoring.

11.1.1 Contractor delivers Soil samples to monitoring personnel with proper chain of custody documentation.

11.1.2 Place up to six samples in a heated sample box. Open bags and remove sample jar lids, Insert temperature probe into a selected soil sample. Close sample box lid and allow samples to equilibrate at 90 ± 10 degrees Fahrenheit for 15 minutes.

11.1.3 Insert MINICAMS probe into the heated sample box. Monitor headspace for two complete cycles on the MINICAMS.

a. If MINICAMS result is below the alarm set point, go to step 11.1.12.

b. If MINICAMS result is above the alarm set point, go to step 11.1.4.

11.1.4 Don Mask and Gloves.

11.1.5 Open sample box and replace lids on sample canisters and close bags.

11.1.6 Allow sample box to re-equilibrate for 15 minutes.

11.1.7 Insert MINICAMS probe into the heated sample box and monitor headspace. Once the MINICAMS result is below the alarm set point, go to step 8.

11.1.8 Don mask and gloves and open lid of sample box. Samples will be monitored one at a time.

11.1.9 Open bag and remove lid from a single sample container. Insert temperature probe into soil sample. Close sample box lid and allow sample to equilibrate at 90 $\pm$ 10 degrees Fahrenheit for 15 minutes.

11.1.10 Insert MINICAMS probe into the heated sample box. Monitor headspace for two complete cycles on the MINICAMS.

a. If MINICAMS result is below the alarm set point, go to step 11.1. 9 for next sample container.

b. If MINICAMS result is above the alarm set point for agent, sample must be confirmed with DAAMS tubes. Go to step 11.1.11.

NOTE. Before samples can be cleared by MINICAMS, Perform a successful 1ul challenge for all agents of interest. If the 1ul challenge fails, perform an additional 1ul challenge. If the 2nd challenge fails, the samples cannot be cleared. Steps 11.1.2 thru 11.1.10 may be performed again to clear the samples after the MINICAMS has been successfully re-calibrated in accordance with sections 5,6,7 or 8 above as appropriate.

11.1.11 Allow samples to re-equilibrate at 90 $\pm$ 10 degrees Fahrenheit for 15 minutes. Collect DAAMS tubes at 400 milliliters per minute for 60 minutes. Transport DAAMS tubes to mobile analytical platform for analysis.

a. If agent is detected from DAAMS tubes analysis, the SSO must be immediately notified.

b. If DAAMS analysis results in a non-detect for agent, go to step 11.1.12.

11.1.12 Give clear samples to the contractor for proper disposition.

11.2 Scrap/PPE/Bulk Item Headspace Monitoring.

11.2.1 A sample of scrap, PPE, or a bulk item must be contained in an environment heated to a minimum of 70 F for 4 hours prior to monitoring with the MINICAMS/DAAMS. The item may be monitored at temperatures of at least 50 F if the item is under full sunlight. However, the use of a heated box may be used to raise the temperature of the items in the case of inclement weather.

11.2.2 Once the item has been contained for 4 hours and meets the referenced temperatures, monitoring may proceed following the MINICAMS/DAAMS procedures in the "Soil Headspace Monitoring" section. It is important to note that the heated box will not be

routinely utilized for scrap/PPE/bulk item monitoring. It is recommended the DAAMS collection procedures be used for items suspected of CWM contamination.

11.2.3 Soil samples are delivered by contractor to monitoring personnel.

11.2.4 Place up to three (3) samples in a sample box heated to 90° ± 10 degrees Fahrenheit. Open bags and remove sample jar lids, close sample box lid and allow samples to equilibrate for 15 minutes.

11.2.5 Connect MINICAMS probe to sample port of the heated sample box and run two complete cycles on the MINICAMS.

a. If MINICAMS reading is below the AEL (clear for agent), go to step 16.

b. If MINICAMS reading is above the AEL (hot for agent), go to step 11.2.6.

11.2.6 Don mask and gloves.

11.2.7 Open sample box and replace lids on sample containers.

11.2.8 Allow samples to re-equilibrate for 15 minutes.

11.2.9 Connect MINICAMS to sample port of heated sample box and resample 2 more complete cycles. If MINICAMS reading is still above the AEL (hot for agent), go to step 11.2.10.

11.2.10 Don mask and gloves, open lid of sample box. Samples will be monitored one at a time.

11.2.11 Remove lid from sample container. Insert MINICAMS probe and run one cycle on the MINICAMS. Close the sample jar and repeat this procedure for each sample. When all samples have been monitored, segregate the samples giving a "Hot" response. If no "Hot" samples are identified, go to step 11.2.13.

11.2.12 Collect DAAMS tubes on the hot samples. Collect tubes at 500 milliliters per minute for 30 minutes. Give tubes to the monitoring technician for analysis.

11.2.13 Place the remaining split samples in the heated sample box.

11.2.14 Allow samples to re-equilibrate for 15 minutes.

11.2.15 Connect MINICAMS to sample port of heated sample box until a clear reading is obtained on the MINICAMS. If samples will not clear after 2 complete cycles, go to step 11.2.16.

11.2.16 Don mask and gloves, open lid of sample box. Samples will be monitored one at a time.

11.2.17 Remove lid from sample container. Insert MINICAMS probe and run one cycle on the MINICAMS. Close the sample jar and repeat this procedure for each sample. When all samples have been monitored, segregate the samples giving a Hot response.

11.2.18 Give clear samples to the monitoring technician for proper disposition.

12.0 LIST OF ACRONYMS

Acronym	Definition
ACAMS	Automatic Continuous Air Monitoring System
ACS	American Chemical Society
AEL	Airborne Exposure Limit
AgF	Silver Flouride
AMC	U. S. Army Materiel Command
ANSI	American National Standards Institute
AR	Army Regulation
ASQC	American Society for Quality Control
ATTF	Agent Testing Task Force
CAR	Corrective Action Request
CARC	Chemical Agent Resistant Coating
CASARM	Chemical Agent Standard Analytical Reference Material
CQAT	CASARM Quality Assurance Team
CQAPCAAM	CASARM Quality Assurance Plan for Chemical Agent Air Monitoring
CRL	Certified Reporting Limit
CSM	Chemical Surety Material
CW	Chemical Weapon
DA	Department of Army
DAAMS	Depot Area Air Monitoring Systems
DHHS	Department of Health and Human Services
DL	Detection Limit
DOD	Department of Defense
FAL	Found action level
FC	Found (or measured) concentration
FPD	Flame-photometric detector
GC	Gas chromatograph, gas chromatography
GPL	General population limit
HPD	Hewlett Packard Dynatherm System
HL	Hazard level
IAW	In accordance with
ID	Identification
IDLH	Immediately Dangerous to Life and Health
IOP	Internal Operating Procedure

ISO	International Organization for Standardization
LCL	Lower control limit
LPL	Lower Performance limit
MINICAMS	Miniature Continuous Air Monitoring System
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
P&A	Precision and accuracy (study)
PTP	Proficiency Test Program
QA	Quality Assurance
QAC	Quality Assurance Coordinator
QC	Quality Control
QL	QC sample prepared in the laboratory
QP	QC sample prepared in the laboratory then aspirated with ambient air
RDT&E	Research, development, testing and evaluation
RTM	Real Time Monitor
SCBA	Self contained breathing apparatus
SOP	Standing Operating Procedure
TAL	Target action level
TC	Target (or true) concentration
TMDE	Testing and Measuring Diagnostic Equipment
TWA	Time Weighted Average
UCL	Upper control limit
UIFM	Uncertainty in the found mass
UPL	Upper Performance Limit
USAPMCD	**U.S. Army, Program Manager for Chemical Demilitarization**
USAECBC	**U.S. Army Edgewood Chemical Biological Center.**
Z	Hazard Level

MINICAMS QP Data Sheet

Alarm set point 0.25 0.7 TWA PMT Voltage_____vdc MBFORM-4 Rev4 MARCH 2003

Location_____ Station_____ Position_____ HSL _____

MINICAMS #_____ Agent: HD VX GB GD L GA Reviewed by_____

Target Concentration= 0.25&1.0TWA 'Found Concentration = FC (4ul=1TWA) Agent Std No._____

MinCal Ht:(XSD)HD-250, L-100, (FPD)VX-100 **Performance Limits=+/-50%for 1ul, +/-25% for 4ul**

	Date	Time	Oper	CAL/HT	FC1 FC1&4ul	FC2 FC1&4ul	CA	FC3 FC1&4ul	#	Date	Time	Oper	CAL/HT	FC1 FC1&4ul	FC2 FC1&4ul	CA	FC3 FC1&4ul
1									16								
2									17								
3									18								
4									19								
5									20								
6									21								
7									22								
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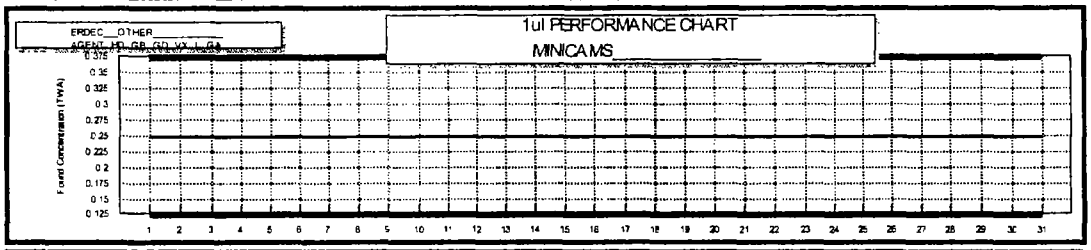
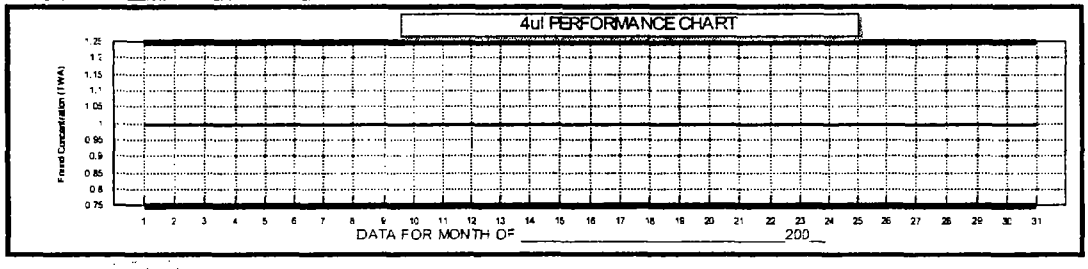
15									30								





(See MT2,9 or16) 'Comments:_____

SAMPLE ONLY

APPENDIX I

14.0 FILLING PROCEDURE FOR GAS STANDARD BAG..

Each operational day the operator shall get either a new or a previously used Tedlar "Gas Standard Bag" that is still functional. If previously used, the operator shall visually check the bag and the "Septa" to assure that the septa or bag will not leak. Note: Leaking bag or Septa will permit the Gas to leak out or permit the intrusion of air into the bag. Either condition will dilute the Gas Standard and cause improper calibration results. **Gas Standard bag size may be either a 1 Litre or a 5 Litre bag depending upon availability and size required.**

Calibration Standard concentrations shall be determined by the team leader, lead chemist, branch chief or operator prior to beginning the bag filling operation to assure that the MINICAMS is calibrated and challenged at the correct TWA levels. The proper calibration and QC standard concentrations shall be calculated based on concentration (gas or liquid) in the tank from which the standard is being taken. If Using the 1000 ml bag, the volume of the standard and the carrier (nitrogen) shall total 1000 ml. e.g If 4 mls of CG is the equivalent of 1 TWA, Then 996 mls of nitrogen shall be added to equal 1000 mls total volume in the bag.

Note: Each sample drawn would then be equivalent to 1 TWA concentration.

1. Purge the 1Litre bag with 500 to 1000 mils of Nitrogen (N2P) prior to filling the gas standard bag. This is applicable to both new and used bags.

2. For 1000 ml bag, fill the bag with 900 mils of Nitrogen and 100 mils of the agent(s) of interest. e.g. 900 mils of Nitrogen & 100 mils CG fills the 1000 ml bag. If using a 5Litre bag (5000 mil bag), the same ratio of Nitrogen to agent shall be maintained.

3. After filling the bag, the operator shall place the bag between the hands and rub the filled bag vigorously by rolling the bag between the hands for approximately 10-15 seconds. This rubbing/rolling operation will remove static and equilibrate the bag temperature while mixing the internal components.

4. If using liquid standards (PS or CF) with gas standards (CG or CK), the Carrier (Nitrogen) and the liquid standards (PS & CF) shall be added together and the bag shal be " Rubbed Vigorously" as in step 2 above before to change the liquids to a gaseous state before adding the gas standards (CG & CK). e.g. Add to bag in the following order: N2P, PS, CF, Rubbing bag vigorously then adding the CG & CK and again rubbing vigoursly.

5. During the MINICAMS Purge Cycle, the filled bag shall be attached to the MINICAMS inlet port by placing the protruding end of the valve into the inlet of the MINICAMS and hand tightening the ¼ inch stainless steel nut with the Teflon ferrule to secure the bag.

6. Before the MINICAMS sample cycle begins, the operator shall manually open the valve on the bag until one (1) complete thread is visible. NOTE: This same procedure shall be used when attaching the filled bag to the end of the "sample line" to permit challenges to be made through the sample line.

7. Bags shall be purged with Nitrogen and "re-filled" for each QP Challenge when challenging every 4--5 hours of operation during personnel safety monitoring.

8. The proper concentration of the filled bag is adversely affected by Heat, Cold and Light so filled bags must be stored and transported inside a closed container that keeps out light and helps maintain a consistent temperature.

NOTE: If several filled bags are required for use during Personnel or Instrument Certification, all bags shall be filled at the same time and stored inside a box to keep out light and maintain a consistent temperature. Each time a bag is used, it should be subjected to the "rubbing" operation before use and placed back in the box after use.

Attachment 7

Internal Operating Procedure and Standing Operation Procedures for Army Analytical Methods

Internal Operating Procedure
Operations Directorate Monitoring Branch

Title: Analysis of Chemical Warfare Agents in Extracts using a Gas Chromatograph/Mass Spectrometer System

Division: Chemical Operations

Branch: Monitoring Branch

Building/Area: E3330 and other areas

This internal operating procedure covers operations, methods and procedures of a general nature not covered by a standing operating procedure. This procedure will be effective until rescinded or superseded. Changes to this procedure will be accomplished by submission of revisions or amendments for approval.

Originator: _____ Date: 10-28-03
Chris V. Giannaras

Analytical Team Leader: _____ Date: 10/28/03
Gail M. Soubie

Quality Assurance Coordinator: _____ Date: 10/28/03
Paul L. Stewart

Monitoring Branch Chief: _____ Date: 10/28/03
Thomas E. Rosso

Date: October 2003
IOP Number: MT-8
Revision Number: 2
Prepared by: C.V. Giannaras (ext. 5-2520)
Approved by: T.E. Rosso (ext. 5-8428)

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Approved by: T.E. Rosso (ext. 5-8428)

RECORD OF CHANGE

Previous revisions of this document did not incorporate a record of change page.

- Revision 0 – IOP created.
- Revision 1 – Table of Contents, Record of Change added
- Revision 2 – Changes made to amount of BFB injected for tuning and combining BFB tune verification with calibration verification. Surrogate and matrix spike compounds and concentrations added. Extract holding time added. Retention time criteria added. Data qualifiers added. Requirement of CCV every 10 samples removed.

Table Of Contents

Analysis of Chemical Warfare Agents in Extracts using a Gas chromatograph/Mass Spectrometer System

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine Chemical Warfare Agents and their breakdown products in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including ground and surface water, aqueous sludges, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following compounds can be determined by this method:

GA, GB, GD, GF, HD, L, HN-1, HN-3, 1,4-Dithiane, 1,4-Thioxane, VX, CEES, TDG, TDP

Other compounds may be determined as long as there is proper demonstration that all quality control criteria can be met.

1.2 The estimated quantitation limit (EQL) for determining an individual compound is approximately 20 - 200 µg/kg (wet weight) for soil/sediment samples and 20 - 200 µg/L for ground water samples. EQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

1.3 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

2.0 ADDITIONAL NON-STANDARD RESPONSIBILITIES

2.1 There are no additional responsibilities for the supervisor, operator, or the supporting activities other than those outlined in Appendix O in the SOP Guidebook.

3.0 APPARATUS AND MATERIALS

3.1 Gas chromatograph/mass spectrometer system

3.1.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the MS source.

3.1.2 Column - 30 m x 0.25 mm, ID 1 µm film thickness, silicone-coated fused-silica capillary column (Restek RTX-1701 or equivalent).

3.1.3 The GC should be equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation.

3.2 Mass spectrometer

3.2.1 Mass spectrometer - Capable of scanning from 35 to 300 amu every 2 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 4-Bromofluorobenzene (BFB) which meets all of the

criteria in Table 2 when 1 - 50 ng of the GC/MS tuning standard (BFB) are injected through the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC.

3.2.2 GC/MS interface - Any GC-to-MS interface may be used that gives acceptable calibration points at 2ng or less per injection for each compound of interest and achieves acceptable tuning performance criteria. For a narrow-bore capillary column, the interface is usually capillary-direct into the mass spectrometer source.

3.3 Data system

3.3.1 Data system - A computer system should be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer should have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software should also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

4.0 HAZARDS

4.1 Dilute Chemical Warfare Agents

4.1.1 The Monitoring Branch utilizes near drinking water level dilute chemical warfare agents in preparing quality control samples for the IOP. The Monitoring Branch office contains a reference file of material safety data sheets (MSDS) for the dilute CW agents.

4.2 Solvents and Other materials.

4.2.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available which includes, but is not limited to, donning the appropriate laboratory safety attire and working in well-ventilated hoods. The laboratory is responsible for maintaining a current file of Occupational Safety and Health Administrations (OSHA) regulations regarding the safe handling of the chemical specified in this IOP. A reference file of material safety data sheets (MSDS) of materials used in these operations is available at the worksite.

5.0 SUMMARY OF METHOD

5.1 The CWAs are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

5.2 Analytes eluted from the capillary column are introduced into the mass spectrometer via direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion to a known amount using a five-point calibration curve.

6.0 PROCEDURE

6.1. Recommended chromatographic conditions

6.1.1 Injector temperature: 250C
6.1.2 Carrier gas (He) flow rate: 1.1 ml/min
6.1.3 Column: Restek RTX-1701, 30m x 0.25 mm
6.1.4 Initial temperature: 60C, hold for 1 minute
6.1.5 Temperature program: 25C/min
6.1.6 Final temperature: 250C, hold for 1.50 minutes

6.2 Recommended MSD conditions

6.2.1 Mass range: 35 - 260 amu (full scan)
6.2.2 Scan time: 0.6 - 2 sec/scan
6.2.3 Source temperature: 150C

6.3 Tuning the MSD

6.3.1 Each GC/MS system must be hardware-tuned to meet the criteria in Table 2 for 1 - 50 ng injection of 4-bromofluorobenzene. Analyses must not begin until these criteria are met.

6.3.1.1 The mass spectrum of BFB must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not background subtract part of the BFB peak.

6.3.1.2 Use the BFB mass intensity criteria in Table 2 as tuning acceptance criteria.

> *NOTE: All subsequent standards, samples, MS/MSDs, LCSs, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.*

6.4 Initial Calibration

6.4.1 Calibration must be performed using the sample introduction technique that will be used for samples.

6.4.2 Proceed with the analysis of the calibration standards following the procedure in the introduction method of choice. For direct injection, inject 1µL – 2 µL into the GC/MS system. The injection volume will depend upon the chromatographic column chosen and the tolerance of the specific GC/MS.

6.4.3 Calibration of an analytical instrument involves the delineation of the relationship between the response of the instrument and the amount or concentration of an analyte introduced into the instrument. The graphical depiction of this relationship is often referred to as the calibration curve. In order to perform quantitative measurements, this relationship must be established prior to the analysis of any samples, and thus, is termed initial calibration. The initial calibration for this chromatographic method involves the analysis of standards containing the target compounds at a minimum of five different concentrations covering the working range of the instrument. In order to produce acceptable sample results, the response of the instrument must be within the working range established by the initial calibration. The extrapolation of the calibration to concentrations above or below those of the actual calibration standards is not appropriate and may lead to significant quantitative errors regardless of the calibration model chosen. Analysts are advised that it may be necessary to prepare calibration standards that cover concentration ranges that are appropriate for specific projects or type of analyses.

6.4.4 For each analyte and surrogate of interest, prepare calibration standards at a minimum of five different concentrations by adding volumes of one or more stock standards and diluting to volume with an appropriate solvent. Typical calibration levels range from 0.05 µg/ml to 20 µg/ml.

6.4.4.1 The lowest concentration calibration standard that is analyzed during an initial calibration establishes the method quantitation limit based on the final volume of extract (or sample) described in the preparative method or employed by the laboratory.

6.4.4.2 The other concentrations should define the linear working range of the detector or correspond to the expected range of concentrations found in actual samples that are also within the working range of the detector.

6.4.4.3 For each analyte, at least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project, which may include establishing compliance with a regulatory or action limit.

6.4.5 External standard calibration procedure

6.4.5.1 External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas are compared to peak areas of the standards. The ratio of the detector response to the amount (mass) of analyte in the calibration standard is defined as the calibration factor (CF).

6.4.5.2 Tabulate the area response of the characteristic ions (see Table 1) against the concentration for each target analyte.

6.4.5.2.1 Linear calibration using a least squares regression

6.4.5.2.1.1 The analyst must employ a regression equation that does not pass through the origin. This is most easily achieved by performing a linear regression of the instrument response versus the concentration of the standards. Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). This is a statistical

requirement and is not simply a graphical convention. For all instances, an appropriate unweighted least squares method should be used. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where:
y = Instrument response (peak area or height)
a = Slope of the line (also called the coefficient of x)
x = Concentration of the calibration standard
b = The intercept

The analyst must not force the line through the origin, but have the intercept calculated from the five data points. The use of a linear regression may not be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards. The regression calculation will generate a correlation coefficient (r^2) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.000 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.990. In calculating sample concentrations by the external standard method, the regression equation is rearranged to solve for the concentration (x), as shown below.

$$x = (y-b)/a$$

6.4.6 Initial calibration verification

6.4.6.1 Immediately following the initial calibration, a calibration verification must be performed (ICV). This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration standard analysis must meet the verification acceptance criteria provided in Sec. 6.5.3.

6.4.6.2 The calibration verification spiking standard must not be prepared from the same standards as the initial calibration standards. However, the same spiking standard prepared for the matrix spike and LCS may be used.

6.5 GC/MS tuning and calibration verification – Tuning verification is performed at the beginning of each 12-hour analytical shift. Calibration verification (CCV) consists of analysis of a midpoint calibration standard from a different source than the intial calibration. It is recommended that additional CCVs be analyzed throughout the sequence (i.e. every 10 samples).

6.5.1 Prior to the analysis of samples or calibration standards, inject or introduce 1 - 50 ng of the 4-bromofluorobenzene standard into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 2 before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

> *NOTE: The BFB and calibration verification (CCV or QC Check) standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.*

6.5.2 The initial calibration curve (Sec. 6.4) for each compound of interest should be verified once every 12 hours prior to sample analysis using the introduction technique used for samples. This is accomplished by analyzing a CCV standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the CCV standard analysis must meet the following verification acceptance criteria: If the percent difference between found and true amounts for each target agent and surrogate is less than or equal to +/-20% (+/-25% for VX and L), the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater than +/-20% difference, +/-25% for VX and L), for any one agent, then corrective action must be taken prior to the analysis of samples. If the agent(s) are not included in the list of analytes for a project, no corrective action is required.

NOTE: If additional CCVs are analyzed and the CCV is greater than +20% (-25% for VX and L), all samples prior to the failing CCV and after the last acceptable CCV must be reanalyzed. If the CCV is greater than +20% (+25% for VX and L), analysis must stop however previous samples may be reported.

6.5.3 The calibration verification spiking (CCV or QC) standard must not be prepared from the same standards as the initial calibration standards. However, the same spiking standard prepared for the matrix spike and LCS may be used.

6.6 A method blank should be analyzed after the calibration standard, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Sec. 7.6.1 for method blank performance criteria.

6.7 GC/MS analysis of samples

6.7.1 BFB tuning criteria and GC/MS calibration verification criteria as describe in sections 6.3, 6.4, and 6.5 must be met before analyzing samples.

6.7.2 All samples and standard solutions must be allowed to warm to ambient temperature before analysis. Set up the introduction device as outlined in the method of choice. Extracts must be analyzed within fourty (40) days from date extraction was completed.

6.7.3 Inject a 1µL - 2µL aliquot of the sample extract into the GC/MS system, using the same operating conditions that were used for the calibration (Sec. 6.4). The injection volume must be the same volume used for the calibration standards.

6.8 Qualitative analysis

6.8.1 The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met.

6.8.2 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

6.8.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

6.9 Quantitative analysis

6.9.1 After a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion.

6.9.2 The concentration in the extract is determined using the calculation in section 6.4.5.2.1.1 from initial calibration data.

6.9.3 If the concentration of any target analyte is below the current laboratory practical reporting limit (PQL) but above the laboratory method detection limit (MDL), the agent concentration is flagged with the "J" qualifier. A concentration below the MDL is consider a non-detect.

6.9.4 If the concentration of any target analyte is above the concentration of the highest level of the initial calibration, a dilution must be performed if there is sufficient sample and the concentration is considered an estimate and flagged with the "E" qualifier. Any concentrations in a dilution analysis must be flagged with the "D" qualifier.

6.9.4.1 The sample should be diluted so that the concentration of the highest agent falls within the range of the mid-level to upper level of the calibration. The amount of dilution, the dilution factor (DF), is calculated as follows:

$$DF = \frac{\text{Highest concentration found}}{\text{concentration of mid-level}}$$

(Round down to the nearest whole number. If greater than 5, round down to nearest multiple of 5.)

For example:

- If GB was detected at 75 ng and the upper calibration range of GB is 5.25 ng, then the extract must be diluted. The mid-level calibration standard for GB is 1.25 ng, therefore the DF will be 60-fold (75/1.25 = 60). If GB was detected at 22.64 ng the DF will be 15-fold (22.64/1.25 = 18.1; round down to nearest 5 = 15)

6.9.5 If a target analyte is detected in any sample and was detected in the method blank, all results are flagged with the "B" qualifier.

7.0 QUALITY CONTROL

7.1 Each laboratory must maintain a formal quality assurance program. The laboratory must also maintain records to document the quality of the data generated.

7.2 The GC/MS system must be tuned to meet the BFB criteria listed in Table 2.

7.3 There must be an initial calibration of the GC/MS system as described in Sec. 6.4.

7.4 The GC/MS system must meet the calibration verification acceptance criteria in Sec. 6.5.

7.4.1 Retention time windows must be establish by the laboratory for each instrument. These windows are determined prior to analysis of samples. The laboratory must calculate new retention time windows for each standard on each GC column and whenever a new GC column is installed. The data must be retained by the laboratory.

7.4.2 A retention time window is calculated for each analyte and surrogate by using the initial calibration check standard (CCV) of each sequence. The windows shall be +/- 0.10 minutes around the absolute retention time for each analyte and surrogate. Analytes are identified when peaks are observed in the RT window for the compound.

7.5 Sample Quality Control for Preparation and Analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank (MB), matrix spike (MS), a duplicate (MSD), a laboratory control sample (LCS), and a duplicate (LCSD) in each analytical batch and the addition of surrogates to each field sample and QC sample.

7.5.1 The suggested surrogate is 4-bromofluorobenzene (BFB). Additional or different surrogates may be used to conform to project specific requirements. The surrogate should be spiked at a level consistant with the mid-point of the instrument calibration range.

7.6 Before processing any samples, the analyst should demonstrate, through the analysis of an instrument blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, an instrument blank should be analyzed as a safeguard against chronic laboratory contamination.

7.6.1 All blanks must be free of target agents or the agents must be below the PQL.

7.7 Documenting the effect of the matrix should include the analysis of one matrix spike (MS)/matrix spike duplicate (MSD) pair and one laboratory control sample (LCS)/laboratory control sample duplicate (LCSD) pair.

7.7.1 The following compounds should be used in the MS/MSD and LCS/LCSD QC samples: 1,4-Dithiane, 1,4-Thioxane, and HD. Additional or different compounds may be used to conform to project specific requirements. These compounds should be spiked at a level consistant with the mid-point of the calibration range.

7.7.2 The spiking solutions should not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

7.7.3 Great care must be taken to maintain the integrity of all standard solutions. It is recommended all standards be stored at minus 4C or less, in amber bottles with PTFE-lined screw-caps.

7.7.4 A Laboratory Control Sample (LCS) and duplicate (LCSD) must be included with each analytical batch. The LCS/LCSD consist of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. When the results of the matrix spike analysis indicate a potential problem because of the sample matrix itself, the LCS/LCSD results are used to verify that the laboratory can perform the analysis in a clean matrix. The average and range of the percent recoveries will be maintained and plotted.

7.7.4.1 All recoveries of the LCS and LCSD must be within current QC limits for recovery (%Rec.) and relative percent difference (RPD).

7.7.4.1.1 The RPD is calculated as follows:

$$RPD = \frac{|LCSA - LCSDA|}{½(LCSA + LCSDA)} \times 100$$

where:

LCSA = amount detected in the LCS
LCSD = amount detected in the LCSD

7.7.4.2 If any recovery fails to meet its criterion, the failing extract must be analyzed again. If the LCS or LCSD fails yet again, it is deemed out-of-control and the entire batch must be re-extracted and re-analyzed.

7.7.4.3 All averages and RPDs should be within current control limits. Out-of-Statistical-Control is defined as one or more of the following:

- An average that is outside either the upper or lower control limit.
- A RPD value that is above the upper control limit.
- Eight (8) or more successive average recovery points on the same side of the central line.

7.7.4.4 If an out-of-statistical-control situation occurs, the analyst must examine the analytical system for unusual conditions and should perform routine maintenance as needed in an attempt to determine the cause of the situation. Recalibration of the analytical system may be required.

7.8 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. The surrogate recovery must meet the criteria describe in Appendix I.

7.9 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc.

7.10 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

7.11 A summary of all quality control criteria is listed in Appendix II.

8.0 REPORTING REQUIREMENTS AND ORDER OF DATA DELIVERABLES

8.1 The analyst shall provide reports and other deliverables as specified in this section unless superceded in writing by a client-specified format. The required content and form of each deliverable is described in this section. All reports and documentation must be:

- Legible
- Clearly labeled and completed in accordance with instructions in this section
- Arranged in the order specified in this section
- Copies must be legible
- Handwritten corrections of the information must be legible, initialed, and dated.
- White-out and Post-It® notes are not allowed

8.1.1 Prior to submission, the analyst shall arrange items and the components of each item in the order listed in these sections.

8.2 The Sample Data Package is divided into the eight major units described in this section. The Sample Data Package shall include data for the analyses of all samples in one Sample Delivery Group (SDG), including field samples, dilutions, re-analyses, blanks, Laboratory Control Samples/ Laboratory Control Samples Duplicate(s) (LCS/LCSD), and any requested or required Matrix Spike/Matrix Spike Duplicate(s) (MS/MSD). A blank, colored (preferably blue) sheet of paper will separate sections.

8.2.1 Section 1

8.2.1.1 Narrative: This document shall be clearly labeled "ECBC Monitoring Branch Analytical Narrative" and shall contain at a minimum:

- Laboratory name
- Client/Project name
- ECBC sample numbers in the SDG
- SDG number
- Batch number(s)

- Detailed documentation of any quality control, sample, shipment and/or analytical problems encountered in processing the samples reported in the data package.
- Discussion of any IOP modification/variance.

8.2.1.2 Injection log covering the entire analytical sequence. If more than a single log is necessary, forms shall be arranged in chronological order by instrument.

8.2.1.3 Scratch Log complete with analyst, instrument, date extracted, date analyzed, and sample results. After supervisor review, this is moved to Section 8 and the clearance log is placed in this section.

8.2.1.4 QC Charts of average LCS %Rec. and RPD for each target analyte.

8.2.1.5 Surrogate Recovery Form. This form includes all QC, samples, dilution, and re-extractions.

8.2.1.6 Method Blank Summary. This form includes all QC, samples, dilution, and re-extractions associated with a method blank. If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank, by instrument.

8.2.2 Section 2

8.2.2.1 GC/MS tune verification. This form is used to report the results of the GC/MS instrument tune verification. For each ion listed on the form, report the percent relative abundance.

> *NOTE: For BFB, one or more of the high mass ions may exceed the abundance of the ion listed on the form as the nominal base peak, m/z 95 for BFB. Despite this possibility, all ion abundances shall be normalized to the nominal base peak listed.*

If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.

8.2.2.2 Initial calibration data, area report, and plots of linear regression shall be included in chronological order by instrument, if more than one instrument is used. Required items are standard(s) quantitation reports and reconstructed ion chromatograms for the initial (five-point) calibration if the initial calibration was performed in the same analytical sequence. If no initial calibration was performed, then only the area report and plots for the current calibration are required. Spectra are not required, however, EICPs displaying each manual integration are (Section 8.3.1).

8.2.3 Section 3

8.2.3.1 Check Standards data and evaluation report shall be included in chronological order by instrument, if more than one instrument is used. The Check Standard Evaluation form must be followed by the quantitation report and the reconstructed ion chromatograms. Spectra are not required, however, EICPs displaying each manual integration are (Section 8.3.1).

8.2.4 Section 4

8.2.4.1 Matrix Spike/Matrix Spike Duplicate Recovery (MS/MSD) data and evaluation form. If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the MS/MSDs, by instrument. The MS/MSD form must be followed by the quantitation report and the reconstructed ion chromatograms. Spectra are not required, however, EICPs displaying each manual integration are (Section 8.3.1).

8.2.5 Section 5

8.2.5.1 Method, reagent, and instrument blanks in chronological order, by instrument. All false positive detections must be Q-deleted. The quantitation report and the reconstructed ion chromatograms plus spectra for all detected and non-detected agents shall be provided.

8.2.6 Section 6

8.2.6.1 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) data and evaluation form. If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank, by instrument. The LCS/LCSD form must be followed by the quantitation report and the reconstructed ion chromatograms. Spectra are not required, however, EICPs displaying each manual integration are (Section 8.3.1).

8.2.7 Section 7

8.2.7.1 Sample data, including dilutions, and re-analyses data, shall be arranged in packets with the quantitation report and the reconstructed ion chromatograms plus spectra for all detected and non-detected agents. The samples shall be placed in increasing Monitoring Branch sample number order, considering both letters and numbers. This may not correspond to the order in which the samples were analyzed.

8.2.8 Section 8

8.2.8.1 Chain of Custodies, copy of standard logbook pages of all standards used in the analyses, and copy of extraction log book showing all samples in the SDG.

8.2.8.2 Any other supporting data. For example, screening data, memos, notations.

8.3 Data and Report Formats

8.3.1 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS system must identify such edits or manual procedures. A hardcopy printout of the Extracted Ion Current Profile (EICP) of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all target agents and surrogates.

8.3.2 The analyst shall identify all samples, including dilutions and re-analyses, Laboratory Control Sample(s)/ Laboratory Control Sample Duplicate(s), Matrix Spike/Matrix Spike Duplicate(s) (MS/MSD), blanks, and standards with a unique Monitoring Branch sample number.

8.3.2.1 For field samples, the Monitoring Branch sample number is the XXX digit unique identifying number assigned at log-in to the MUD tracking system. In order to facilitate data assessment, the analyst shall use the following sample suffixes:

- XXXXX = Monitoring Branch sample number
- XXXXXMS = Matrix spike sample
- XXXXXMSD = Matrix spike duplicate sample
- XXXXXRE = Re-extracted and re-analyzed sample
- XXXXXDL = Sample analyzed at a dilution
- XXXXXDL2 = Sample analyzed at a secondary dilution
- XXXXXDL3 = Sample analyzed at a third dilution
- XXXXXDUP = Sample duplicate

8.3.2.2 The Monitoring Branch sample number shall be unique for each blank and LCS/LCSD within an SDG. The unique number will be the eight digit extraction batch number (as described in SOP#XXX) plus the following suffixes:

- XXXXXXXX = extraction batch number
- XXXXXXXXMB = Method blank
- XXXXXXXXLCS = Laboratory control sample
- XXXXXXXXLCSD = Laboratory control sample duplicate

8.3.3 Cross out unused columns and spaces. Initial and date all cross outs.

8.3.4 Do not use paper clips or staple pages together.

8.3.5 A summary of the data format and order can be found in Appendix III.

TABLE 1
CHARACTERISTIC MASSES (m/z) FOR CHEMICAL WARFARE AGENTS

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
GA	106	117, 133
GB	125	99, 81
GD (1 & 2)	126	99, 82
GF	99	67, 54
HD	158	160, 109
HN-1	120	122, 92
HN-3	154	156, 63
L	212	151, 86
VX	114	127, 167
1,4-Thioxane	104	61, 74
1,4-Dithiane	120	127, 167
CEES	124	75, 126
TDG	104	61, 45
TDP	150	61, 93

TABLE 2
BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA

m/z	Required Intensity (relative abundance)
50m/z	8 to 40% of m/z 95
75m/z	30 to 66% of m/z 95
95m/z	Base peak, 100% relative abundance
174m/z	Greater than 50% to 120% of m/z 95
176m/z	Greater than 93% but less than 101% of m/z 174

Appendix I

It is necessary that the laboratory evaluate surrogate recovery data from individual samples. The analyst must evaluate the recovery to determine if acceptable system performance was maintained for each analysis. Surrogate limits are established by the laboratory annually, or as specified by clients or programs. The surrogate recovery is calculated as:

$$\textit{Recovery (\%)} = \frac{\textit{Concentration (or amount) found}}{\textit{Concentration (or amount) added}} \times 100$$

If the recovery is not within surrogate recovery limits, the following procedures are necessary:

- Check to be sure that there are no errors in the calculations or surrogate solution standard. If errors are found, recalculate the data accordingly.
- Rerun the extract (to ensure a proper injection). If it fails again, the sample must be re-extracted (in a new batch with new QC) and re-analyzed.
- If the re-extraction passes, then it is assumed there was a spiking error and the initial analysis is not reported. If it fails yet again, then it is assumed there is a matrix affect and both (the initial and re-extraction) analyses are reported.

Appendix II

Typical analytical sequence

With initial calibration:
1. tune with 1 - 50ng BFB
2. instrument blank
3. 5-point calibration
4. calibration verification
5. method blank
6. LCS/LCSD
7. samples, MS/MSD
8. CCV (every 10 samples)
9. instrument blank (optional)
10 ending calibration check standard

Without initial calibration:
1. tune with 1 - 50ng BFB
2. instrument blank
3. calibration verification
4. method blank
5. LCS/LCSD
6. samples, MS/MSD
7. CCV (every 10 samples)
8. instrument blank (optional)
9. ending calibration check standard

QC Criteria

- Tune: GC/MS system must meet all criteria before any analysis (Table 2).
- Initial Calibration: linear regression (do not force zero). Correlation coefficient (r^2) ≥ 0.990 (section 6.4.5.2.1.1).
- Calibration verification/check: +/- 20% (25% for VX and L) difference (section 6.5.2).
    - If calibration check is less that 20% (25% for VX and L), all samples prior to the calibration check must be reanalyzed. If the calibration check is greater than 20% (25% for VX and L), analysis must stop however previous samples maybe reported.
- Surrogate: all surrogate recoveries must be within current lab limits. If not, sample must be re-analyzed. If it fails again, the sample must be re-extracted and re-analyzed (Appendix I).
- LCS/LCSD: %Rec. and RPD must be within current lab limits. If not, re-analyze. If either one fails again, the entire batch must be re-extracted. Sample analysis can not begin until the LCS/LCSD meet all criteria (section 7.7.4).
- MS/MSD: recoveries are evaluated against current laboratory limits, however, if any recovery is outside these limits there is no correction action. All failures will be discussed in the report narrative.

Appendix III
Data Package Format

- Section 1
  - Narrative
  - Checklist
  - Scratch log
  - Injection log
  - QC Charts
  - Surrogate summary
  - Method Blank summary

- Section 2 (Calibration)
  - Tune verification data in chronological order
  - Initial Calibration data in chronological order, by instrument
    - Area report and plots
    - Raw data(on day that a calibration is analyzed – no spectra except for manual integrations)

- Section 3 (Check standards)
  - Check standards in chronological order
    - Check standard evaluation report
    - Quant report/chromatogram(no spectra except for manual integrations)

- Section 4 (MS/MSD)
  - MS/MSDs in chronological order, by instrument
    - MS/MSD evaluation report
    - Quant report/chromatogram(no spectra except for manual integrations)

- Section 5 (Blanks)
  - Blanks in chronological order
  - Detailed report for each blank

- Section 6 (LCS/LCSD)
  - LCS/LCSDs in chronological order, by instrument
    - LCS/LCSD evaluation report
    - Quant report/chromatogram(no spectra except for manual integrations)

- Section 7 (Analyses – in Monitoring Branch sample # order)
- Quant report/ Chromatogram (with label)
    - Final quant report (after Qdeleting false hits)
    - Manual spectra/blowups of target agents

- Section 8 (support data)
  - COCs
  - Logbook pages (extraction/standards)
  - Any other information (i.e. memos, notes, support data)

STANDING OPERATING PROCEDURE

Building(s): E3330, E3832 & other OCONUS/CONUS locations

Room #s: 186 (E3330)

(check all that apply)

_____Chemical Agent
_____Biological/Toxin
_____Radiation
_____Pyrotechnic
XXXX Other (specify: dilute agent)

ECBC
CB SERVICES DIRECTORATE

Title:
Extraction for Trace Levels of Chemical Warfare Agents, Degradation Products, and Industrial Compounds in Various Environmental Samples

SOP#: CR4-2NP001

Submitted by:

_____
Team Leader/Division Chief

Environmental Quality Office: _____

Risk Reduction Office: _____

Approved by:

_____
Director

APPROVAL DATE: _____

Prepared by: AMSSB-RCB-CM/Tom Rosso

SOP TITLE: Extraction for Trace Levels of Chemical Warfare Agents, Degradation Products and Industrial Compounds in Various Environmental Samples

Operator's Statement: I have read, or have had read to me, the procedures in this SOP and ERDEC-SP-058, Preparation of Standing Operating Procedures. I, by my signature below, indicate that I thoroughly understand and agree to abide by these instructions.

Name (print)	Signature	Date

Supervisor's Statement: I have personally reviewed this IOP and, to the best of my knowledge, believe that the information listed herein is correct.

Supervisor:_____ Date:_____
Thomas E. Rosso

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine Chemical Warfare Agents, breakdown products, and industrial compounds in a variety of matrices. This method is applicable to nearly all types of samples, regardless of water content, including ground and surface water, aqueous sludges, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following compounds can be determined by this method:

GA, GB, GD, GF, HD, L, HN-1, HN-3, 1,4-Dithiane, 1,4-Thioxane, VX, CEES, TDG, TDP

Other compounds may be determined as long as there is proper demonstration that all quality control criteria can be met. The procedures listed in this SOP will also be utilized for the preparation of sample extracts and the quality control sample extracts necessary to complete the analysis for lewisite and derivatized compounds.

2.0 ADDITIONAL NON-STANDARD RESPONSIBILITIES

There are no additional responsibilities for the supervisor, operator, or the supporting activities other than those outlined in Appendix O in the SOP Guidebook.

3.0 MATERIALS

3.1 Dilute Chemical Agent Standards: All dilute agents standards used in this SOP are prepared prior to beginning the operation. The standards shall be maintained in a GC vial with septum cap. The GC vial shall contain no more than 1 milliliter of solution. One or all of these chemicals may be used in this SOP.

3.2 Dilute CW degradation standards: Other compounds may be added under the guidance of an experienced operator.

3.3 Various Organic Solvents and Other Reagents including

3.3.1 Methylene Chloride
3.3.2 Acetone
3.3.3 Hexane
3.3.4 Methanol
3.3.5 Isopropyl Alcohol
3.3.6 Beta-mercaptoethanol (BME)
3.3.7 Ethyl Acetate
3.3.8 4-Bromofluorobenzene (BFB)
3.3.9 Diisopropylaminoethanol (DIPAE)
3.3.10 Sodium Sulfate

4.0 HAZARDS

4.1 Dilute Chemical Warfare Agents

4.1.1 The Monitoring Branch utilizes near drinking water level dilute chemical warfare agents in preparing quality control samples for the SOP. The Monitoring Branch office contains a reference file of material safety data sheets (MSDS) for the dilute CW agents.

4.2 Solvents and Other materials

4.2.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. This includes, but is not limited to, donning the appropriate laboratory safety attire and working in well-ventilated hoods. The laboratory is responsible for maintaining a current file of Occupational Safety and Health Administrations (OSHA) regulations regarding the safe handling of the chemical specified in this SOP. A reference file of material safety data sheets (MSDS) of materials used in these operations is available at the worksite.

5.0 SAFETY REQUIREMENTS

5.1 Ventilation

5.1.1 Laboratory hoods when used in this operation shall be certified for use through the Hazards Surveillance Office (HSO) prior to initiation of operation.

5.1.2 Pre-Operative checks of the face velocity of the hood to assure compliance with the average face velocity noted on the hood certification sticker shall be accomplished by operating personnel. If the average of 3 vaneometer readings deviates by more than 20% of the average face velocity on the certification sticker, then do not begin operations. Notify the ECBC Safety Office immediately and contact the Hazards Surveillance Office for recertification of the hood.

5.2 Monitoring

5.2.1 No monitoring is necessary during this SOP operation.

5.3 Protective Clothing

5.3.1 Surgical or latex gloves must be used and discarded after each operation. Users of surgical or latex gloves must also avoid sources of ignition.

5.3.2 Chemical safety glasses will be worn.

5.3.3 A lab coat or uniform will be worn.

5.4 Pre-Operational Checklist

5.4.1 The Pre-Operational Checklist, see Appendix I, must be completed and signed by the primary analyst and his/her buddy before proceeding with the extraction process.

6.0 SUMMARY OF METHOD

6.1 Chemical warfare agents are extracted from various matrices using organic solvents. The extract is retained and analyzed via GC/MS.

7.0 PROCEDURES

7.1 Sample Receipt

7.1.1 Receive samples from CTF, contractor personnel on-site, or accept cooler from courier.

7.1.2 Verify contents against supplied chain of custody. Note any discrepancies or variances. Sign and date chain of custody documentation.

7.1.3 If supplied, check and document the temperature of the temperature blank on COC and in the extractions log.

7.2 Extract Sample

NOTE: The following sections describe the extraction of samples, laboratory QC, and field QC. The typical weights and volumes are listed, however circumstances and/or project requirements may dictate different weights and volumes. A 1:1 ratio of sample to total solvent volume is required to maintain reporting limits. Any other ratio will affect the reporting limits and require adjustments to any detected compound. For example, 2 gm of sample extracted with a total solvent volume (solvent vol., surrogate vol., spike vol.) of 2ml is the same as 1gm of sample extracted with a total solvent volume of 1ml. If 1gm of soil was extracted with a total solvent volume of 2ml, the reporting limits would increase by 2-fold and any detected compounds would need to be multiplied by two.

7.2.1 Don appropriate personnel protective equipment (PPE).

7.2.2 Weigh out approximately 2 grams or 2 ml of sample (record actual weight in extractions logbook) into a glass or polypropylene tube.

7.2.3 Spike with 100 ul of 20 ng/ul (ppm) surrogate BFB standard. Record the exact volume spiked in the extraction log book.

7.2.4 Add 1.9 ml of the appropriate extraction solvent (Table 1) using a solvent dispenser. Record the exact volume used in the extraction log book.

7.2.5 Vortex the sample tube for approximately 10 seconds and allow the solvent layer to separate. Transfer extract into syringe. For liquid samples, transfer the bottom layer.

7.2.6 Attach a 0.45 µm pore size PTFE filter to the syringe. Gently push the barrel of the syringe to force the extract through the filter. Capture the filtered extract in a labeled autosampler vial. Seal vial. The sample is ready for analysis.

7.3. Quality Control Samples

7.3.1 Method blank

7.3.1.1 Prepare a method blank at the same time samples are being extracted with every extraction batch (Section 9.0). Use sodium sulfate for soil simulant and distilled water for aqueous sample simulant and prepare as in section 7.2

7.3.2 Laboratory Control Samples (LCS/LCSD)

7.3.2.1 Prepare a laboratory control sample (LCS) and duplicate (LCSD) at the same time samples are being extracted with every extraction batch. Use sodium sulfate for soil simulant and distilled water for aqueous sample simulant and prepare as in section 7.2.2

7.3.2.2 Spike with 100 ul of 20 ng/ul (ppm) surrogate BFB standard. Record the exact volume spiked in the extraction logbook.

7.3.2.3 Spike with 100 ul of each matrix spike solution. Record the exact volume spiked in the extraction logbook.

7.3.2.4 Add enough of the appropriate extraction solvent (Table 1) to reach a final volume of 2.0 ml using a solvent dispenser. Record the exact volume added in the extraction logbook.
7.3.2.5 Vortex the sample tube for approximately 10 seconds and allow the solvent layer to separate. Transfer extract into syringe. For liquid samples, transfer the bottom layer.

7.3.2.6 Attach a 0.45 µm pore size PTFE filter to the syringe. Gently push the barrel of the syringe to force the extract through the filter. Capture the filtered extract in a labeled autosampler vial. Seal vial. The sample is ready for analysis.

7.3.2.7 Repeat all steps in section 7.3.2 for laboratory control sample duplicate (LCSD)

7.3.3 Matrix Spike Samples (MS/MSD)

7.3.3.1 Prepare additional samples for matrix spike (MS) and matrix spike duplicate (MSD) analysis for every batch.

7.3.3.2 Weigh out approximately 2 grams or 2ml of sample (record actual weight in extractions logbook) into a glass or polypropylene tube.

7.3.3.3 Spike with 100 ul of 20 ng/ul (ppm) surrogate BFB standard. Record the exact volume spiked in the extraction log book.

7.3.3.4 Spike with 100 ul of each matrix spike solution. Record the exact volume spiked in the extraction log book.

7.3.3.5 Add enough of the appropriate extraction solvent (Table 1) to reach a final volume of 2.0 ml using a solvent dispenser. Record the volume added in the extraction log book.

7.3.3.6 Vortex the sample tube for approximately 10 seconds and allow the solvent layer to separate. Transfer extract into syringe. For liquid samples, transfer the bottom layer.

7.3.3.7 Attach a 0.45 µm pore size PTFE filter to the syringe. Gently push the barrel of the syringe to force the extract through the filter. Capture the filtered extract in a labeled autosampler vial. Seal vial. The sample is ready for analysis.

7.3.3.8 Repeat all steps in section 7.3.3 for matrix spike duplicate.

7.4 Environmental Management

7.4.1 After samples are analyzed, remaining sample (solid or liquid) will be managed as hazardous waste. If liquid remains in sample container, it will be added to decontamination bath and carry an

appropriate hazardous waste code. If any solid sample remains, it will be added to hazardous waste container and carry an appropriate hazardous waste code.

7.4.2 For general disposal procedures, refer to ERDEC-SP-058, Appendix M, Environmental Considerations. For specific disposal procedures regarding this SOP, refer to Appendix II. All waste will be managed IAW APGR 200-60.

8.0 EMERGENCY AND FIRST AID PROCEDURES.

First Aid and Emergency Procedures for the chemicals involved in this operation are described in the individual MSDS sheets. In the event of an emergency, Dial 911 and also notify appropriate chain of command to include the Risk Reduction Office (x5-4414/4411) within one hour of the emergency.

9.0 BATCH DEFINITION

9.1 An extraction batch is defined as "samples of the same matrix processed together at the same time." The extraction batch size is twenty (20) field samples or less. Laboratory control samples (method blank, LCS, LCSD) are not counted in the 20 sample limit but MS and MSDs are counted.

9.2 Quality Control Samples

9.2.1 Each extraction batch must contain a method blank, laboratory control sample (LCS), and laboratory control sample duplicate (LCSD). These samples are required to measure the laboratory's ability to successfully extract target agents under controlled conditions.

9.2.2 Each extraction batch must contain a matrix spike (MS) and matrix spike duplicate (MSD). This consists of a field sample spiked with known amounts of the target agents. These are required to measure any presence of a matrix affect.

9.3 Batch Number

9.3.1 A unique batch number will be generated for every extraction batch. The batch number will be in the following format:

- yymmddxx

Where:

- yymmdd = date the extraction was performed
- xx = sequential numeric suffix representing each batch analyzed on that date

TABLE 1

EXTRACTION SOLVENTS

Agent	Extraction Solvent
Any agents and VX (no L)	CH_2Cl_2 fortified with 0.01% DIPAE
Any agents and L (no VX)	CH_2Cl_2 fortified with 0.05% BME
TDG	Ethyl Acetate
Any agent/compound (except VX, L, or TDG)	CH_2Cl_2

Note: For samples requiring VX and L or any agent and TDG, separate extractions are needed. These will be in unique batches, therefore each will contain it's own unique QC samples (MB, LCS, LCSD, MS, MSD).

Appendix I

SOP Pre-Operational Checklist

SOP#:_____ Date:_____

Building/Location:_____ Room:_____

The operator's buddy will be in the work area prior to the operation startup. All operators, primary and buddy, will check, sign, and date this pre-operational checklist before each operation.

	YES	NO
1. Ventilation/Hood velocity checked	_____	_____
2. SOP on site	_____	_____
3. Sufficient DECON available	_____	_____
4. Protective clothing (gloves, lab coat, safety glasses)	_____	_____
5. Operators:		
a. Medically cleared	_____	_____
b. Read and signed SOP	_____	_____
c. Hazardous waste requirements	_____	_____
d. Reportable sores/abrasions	_____	_____
6. Eyewash available	_____	_____
7. Emergency shower available	_____	_____

Primary Operator:_____ Secondary Operator:_____
(print) (print)

Primary Operator:_____ Secondary Operator:_____
(signature) (signature)

Appendix II

Environmental Considerations

Type of material generated and amount (if known)	1) Gloves, syringe filter, environmental sample, glass/polypropylene tube 2) Extraction solvents/Reagents	1) Decon solution of environmental samples 2) Excess extracts 3) Reagents	N/A
RCRA hazardous/non-hazardous/agent contaminated determination (if RCRA hazardous, identify corrosive, TCLP, reactive, ignitable, or listed)	1) Non-Regulated (if deconned) 2) When CH2Cl2 is solvent, remaining solid waste will be F002. When ethyl acetate is solvent, remaining solid waste will be F003.	1) Non-regulated for decon solution. 2) When CH2Cl2 is solvent, F002 applies to the waste. When ethyl acetate is solvent, F003 applies to the waste. 3) Some of the reagents are flammable under certain conditions, and would carry a D001 waste code. They should be treated as hazardous waste.	N/A
Ultimate disposal methods(describe in detail)	Dispose of IAW APG-R 200-60 via the hazardous waste tracking system	1) Will be turned in via HWTS IAW APGR 200-60. 2&3) Disposed of as Hazardous waste IAW APGR 200-60	N/A
Spill control method	N/A	Secondary containment	N/A
Spill material disposal method	Same as above for ultimate disposal	Spill sorbed on sorbant, swept up, and placed in fiber drums for disposal. Will be turned-in IAW APGR 200-60.	N/A

Internal Operating Procedure
Operations Directorate Monitoring Branch

Title: Analysis of Chemical Warfare Agents in Extracts using a Gas Chromatograph/Mass Spectrometer System

Division: Chemical Operations

Branch: Monitoring Branch

Building/Area: E3330 and other areas

This internal operating procedure covers operations, methods and procedures of a general nature not covered by a standing operating procedure. This procedure will be effective until rescinded or superseded. Changes to this procedure will be accomplished by submission of revisions or amendments for approval.

Originator: _____ Date: 10-28-03
Chris V. Giannaras

Analytical Team Leader: _____ Date: 10/28/03
Gail M. Soubie

Quality Assurance Coordinator: _____ Date: 10/28/03
Paul L. Stewart

Monitoring Branch Chief: _____ Date: 10/28/03
Thomas E. Rosso

Date: October 2003
IOP Number: MT-8
Revision Number: 2
Prepared by: C.V. Giannaras (ext. 5-2520)
Approved by: T.E. Rosso (ext. 5-8428)

Internal Operating Procedure
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This internal operating procedure covers operations, methods and procedures of a general nature not covered by a standing operating procedure. This procedure will be effective until rescinded or superseded. Changes to this procedure will be accomplished by submission of revisions or amendments for approval.

Originator:_____ Date:_____
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Thomas E. Rosso

Date: October 2003
IOP Number: MT-8
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Prepared by: C.V. Giannaras (ext. 5-2520)
Approved by: T.E. Rosso (ext. 5-8428)

RECORD OF CHANGE

Previous revisions of this document did not incorporate a record of change page.

- Revision 0 – IOP created.
- Revision 1 – Table of Contents, Record of Change added
- Revision 2 – Changes made to amount of BFB injected for tuning and combining BFB tune verification with calibration verification. Surrogate and matrix spike compounds and concentrations added. Extract holding time added. Retention time criteria added. Data qualifiers added. Requirement of CCV every 10 samples removed.

Table Of Contents

Title: Analysis of Chemical Warfare Agents in Extracts using a Gas chromatograph/Mass Spectrometer System

Operator's Statement: I have read, or have had read to me, the procedures in this IOP. I, by my signature below, indicate that I thoroughly understand and agree to abide by these instructions.

Name (print)	Signature	Date

Supervisor's Statement: I have personally reviewed this IOP and, to the best of my knowledge, believe that the information listed herein is correct.

Supervisor:_____ Date:_____

Thomas E. Rosso

Analysis of Chemical Warfare Agents in Extracts using a Gas chromatograph/Mass Spectrometer System

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine Chemical Warfare Agents and their breakdown products in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including ground and surface water, aqueous sludges, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following compounds can be determined by this method:

GA, GB, GD, GF, HD, L, HN-1, HN-3, 1,4-Dithiane, 1,4-Thioxane, VX, CEES, TDG, TDP

Other compounds may be determined as long as there is proper demonstration that all quality control criteria can be met.

1.2 The estimated quantitation limit (EQL) for determining an individual compound is approximately 20 - 200 µg/kg (wet weight) for soil/sediment samples and 20 - 200 µg/L for ground water samples. EQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

1.3 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

2.0 ADDITIONAL NON-STANDARD RESPONSIBILITIES

2.1 There are no additional responsibilities for the supervisor, operator, or the supporting activities other than those outlined in Appendix O in the SOP Guidebook.

3.0 APPARATUS AND MATERIALS

3.1 Gas chromatograph/mass spectrometer system

3.1.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the MS source.

3.1.2 Column - 30 m x 0.25 mm, ID 1 µm film thickness, silicone-coated fused-silica capillary column (Restek RTX-1701 or equivalent).

3.1.3 The GC should be equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation.

3.2 Mass spectrometer

3.2.1 Mass spectrometer - Capable of scanning from 35 to 300 amu every 2 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 4-Bromofluorobenzene (BFB) which meets all of the

criteria in Table 2 when 1 - 50 ng of the GC/MS tuning standard (BFB) are injected through the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC.

3.2.2 GC/MS interface - Any GC-to-MS interface may be used that gives acceptable calibration points at 2ng or less per injection for each compound of interest and achieves acceptable tuning performance criteria. For a narrow-bore capillary column, the interface is usually capillary-direct into the mass spectrometer source.

3.3 Data system

3.3.1 Data system - A computer system should be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer should have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software should also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

4.0 HAZARDS

4.1 Dilute Chemical Warfare Agents

4.1.1 The Monitoring Branch utilizes near drinking water level dilute chemical warfare agents in preparing quality control samples for the IOP. The Monitoring Branch office contains a reference file of material safety data sheets (MSDS) for the dilute CW agents.

4.2 Solvents and Other materials.

4.2.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available which includes, but is not limited to, donning the appropriate laboratory safety attire and working in well-ventilated hoods. The laboratory is responsible for maintaining a current file of Occupational Safety and Health Administrations (OSHA) regulations regarding the safe handling of the chemical specified in this IOP. A reference file of material safety data sheets (MSDS) of materials used in these operations is available at the worksite.

5.0 SUMMARY OF METHOD

5.1 The CWAs are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

5.2 Analytes eluted from the capillary column are introduced into the mass spectrometer via direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion to a known amount using a five-point calibration curve.

6.0 PROCEDURE

6.1. Recommended chromatographic conditions

6.1.1 Injector temperature: 250C
6.1.2 Carrier gas (He) flow rate: 1.1 ml/min
6.1.3 Column: Restek RTX-1701, 30m x 0.25 mm
6.1.4 Initial temperature: 60C, hold for 1 minute
6.1.5 Temperature program: 25C/min
6.1.6 Final temperature: 250C, hold for 1.50 minutes

6.2 Recommended MSD conditions

6.2.1 Mass range: 35 - 260 amu (full scan)
6.2.2 Scan time: 0.6 - 2 sec/scan
6.2.3 Source temperature: 150C

6.3 Tuning the MSD

6.3.1 Each GC/MS system must be hardware-tuned to meet the criteria in Table 2 for 1 - 50 ng injection of 4-bromofluorobenzene. Analyses must not begin until these criteria are met.

6.3.1.1 The mass spectrum of BFB must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not background subtract part of the BFB peak.

6.3.1.2 Use the BFB mass intensity criteria in Table 2 as tuning acceptance criteria.

> *NOTE: All subsequent standards, samples, MS/MSDs, LCSs, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.*

6.4 Initial Calibration

6.4.1 Calibration must be performed using the sample introduction technique that will be used for samples.

6.4.2 Proceed with the analysis of the calibration standards following the procedure in the introduction method of choice. For direct injection, inject 1µL – 2 µL into the GC/MS system. The injection volume will depend upon the chromatographic column chosen and the tolerance of the specific GC/MS.

6.4.3 Calibration of an analytical instrument involves the delineation of the relationship between the response of the instrument and the amount or concentration of an analyte introduced into the instrument. The graphical depiction of this relationship is often referred to as the calibration curve. In order to perform quantitative measurements, this relationship must be established prior to the analysis of any samples, and thus, is termed initial calibration. The initial calibration for this chromatographic method involves the analysis of standards containing the target compounds at a minimum of five different concentrations covering the working range of the instrument. In order to produce acceptable sample results, the response of the instrument must be within the working range established by the initial calibration. The extrapolation of the calibration to concentrations above or below those of the actual calibration standards is not appropriate and may lead to significant quantitative errors regardless of the calibration model chosen. Analysts are advised that it may be necessary to prepare calibration standards that cover concentration ranges that are appropriate for specific projects or type of analyses.

6.4.4 For each analyte and surrogate of interest, prepare calibration standards at a minimum of five different concentrations by adding volumes of one or more stock standards and diluting to volume with an appropriate solvent. Typical calibration levels range from 0.05 µg/ml to 20 µg/ml.

6.4.4.1 The lowest concentration calibration standard that is analyzed during an initial calibration establishes the method quantitation limit based on the final volume of extract (or sample) described in the preparative method or employed by the laboratory.

6.4.4.2 The other concentrations should define the linear working range of the detector or correspond to the expected range of concentrations found in actual samples that are also within the working range of the detector.

6.4.4.3 For each analyte, at least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project, which may include establishing compliance with a regulatory or action limit.

6.4.5 External standard calibration procedure

6.4.5.1 External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas are compared to peak areas of the standards. The ratio of the detector response to the amount (mass) of analyte in the calibration standard is defined as the calibration factor (CF).

6.4.5.2 Tabulate the area response of the characteristic ions (see Table 1) against the concentration for each target analyte.

6.4.5.2.1 Linear calibration using a least squares regression

6.4.5.2.1.1 The analyst must employ a regression equation that does not pass through the origin. This is most easily achieved by performing a linear regression of the instrument response versus the concentration of the standards. Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). This is a statistical

requirement and is not simply a graphical convention. For all instances, an appropriate unweighted least squares method should be used. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where:
y = Instrument response (peak area or height)
a = Slope of the line (also called the coefficient of x)
x = Concentration of the calibration standard
b = The intercept

The analyst must not force the line through the origin, but have the intercept calculated from the five data points. The use of a linear regression may not be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards. The regression calculation will generate a correlation coefficient (r^2) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.000 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.990. In calculating sample concentrations by the external standard method, the regression equation is rearranged to solve for the concentration (x), as shown below.

$$x = (y-b)/a$$

6.4.6 Initial calibration verification

6.4.6.1 Immediately following the initial calibration, a calibration verification must be performed (ICV). This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration standard analysis must meet the verification acceptance criteria provided in Sec. 6.5.3.

6.4.6.2 The calibration verification spiking standard must not be prepared from the same standards as the initial calibration standards. However, the same spiking standard prepared for the matrix spike and LCS may be used.

6.5 GC/MS tuning and calibration verification – Tuning verification is performed at the beginning of each 12-hour analytical shift. Calibration verification (CCV) consists of analysis of a midpoint calibration standard from a different source than the intial calibration. It is recommended that additional CCVs be analyzed throughout the sequence (i.e. every 10 samples).

6.5.1 Prior to the analysis of samples or calibration standards, inject or introduce 1 - 50 ng of the 4-bromofluorobenzene standard into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 2 before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

> *NOTE: The BFB and calibration verification (CCV or QC Check) standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.*

6.5.2 The initial calibration curve (Sec. 6.4) for each compound of interest should be verified once every 12 hours prior to sample analysis using the introduction technique used for samples. This is accomplished by analyzing a CCV standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the CCV standard analysis must meet the following verification acceptance criteria: If the percent difference between found and true amounts for each target agent and surrogate is less than or equal to +/-20% (+/-25% for VX and L), the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater than +/-20% difference, +/-25% for VX and L), for any one agent, then corrective action must be taken prior to the analysis of samples. If the agent(s) are not included in the list of analytes for a project, no corrective action is required.

NOTE: If additional CCVs are analyzed and the CCV is greater than +20% (-25% for VX and L), all samples prior to the failing CCV and after the last acceptable CCV must be reanalyzed. If the CCV is greater than +20% (+25% for VX and L), analysis must stop however previous samples may be reported.

6.5.3 The calibration verification spiking (CCV or QC) standard must not be prepared from the same standards as the initial calibration standards. However, the same spiking standard prepared for the matrix spike and LCS may be used.

6.6 A method blank should be analyzed after the calibration standard, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Sec. 7.6.1 for method blank performance criteria.

6.7 GC/MS analysis of samples

6.7.1 BFB tuning criteria and GC/MS calibration verification criteria as describe in sections 6.3, 6.4, and 6.5 must be met before analyzing samples.

6.7.2 All samples and standard solutions must be allowed to warm to ambient temperature before analysis. Set up the introduction device as outlined in the method of choice. Extracts must be analyzed within fourty (40) days from date extraction was completed.

6.7.3 Inject a 1µL - 2µL aliquot of the sample extract into the GC/MS system, using the same operating conditions that were used for the calibration (Sec. 6.4). The injection volume must be the same volume used for the calibration standards.

6.8 Qualitative analysis

6.8.1 The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met.

6.8.2 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

6.8.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

6.9 Quantitative analysis

6.9.1 After a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion.

6.9.2 The concentration in the extract is determined using the calculation in section 6.4.5.2.1.1 from initial calibration data.

6.9.3 If the concentration of any target analyte is below the current laboratory practical reporting limit (PQL) but above the laboratory method detection limit (MDL), the agent concentration is flagged with the "J" qualifier. A concentration below the MDL is consider a non-detect.

6.9.4 If the concentration of any target analyte is above the concentration of the highest level of the initial calibration, a dilution must be performed if there is sufficient sample and the concentration is considered an estimate and flagged with the "E" qualifier. Any concentrations in a dilution analysis must be flagged with the "D" qualifier.

6.9.4.1 The sample should be diluted so that the concentration of the highest agent falls within the range of the mid-level to upper level of the calibration. The amount of dilution, the dilution factor (DF), is calculated as follows:

$$DF = \frac{\text{Highest concentration found}}{\text{concentration of mid-level}}$$

(Round down to the nearest whole number. If greater than 5, round down to nearest multiple of 5.)

For example:

- If GB was detected at 75 ng and the upper calibration range of GB is 5.25 ng, then the extract must be diluted. The mid-level calibration standard for GB is 1.25 ng, therefore the DF will be 60-fold (75/1.25 = 60). If GB was detected at 22.64 ng the DF will be 15-fold (22.64/1.25 = 18.1; round down to nearest 5 = 15)

6.9.5 If a target analyte is detected in any sample and was detected in the method blank, all results are flagged with the "B" qualifier.

7.0 QUALITY CONTROL

7.1 Each laboratory must maintain a formal quality assurance program. The laboratory must also maintain records to document the quality of the data generated.

7.2 The GC/MS system must be tuned to meet the BFB criteria listed in Table 2.

7.3 There must be an initial calibration of the GC/MS system as described in Sec. 6.4.

7.4 The GC/MS system must meet the calibration verification acceptance criteria in Sec. 6.5.

7.4.1 Retention time windows must be establish by the laboratory for each instrument. These windows are determined prior to analysis of samples. The laboratory must calculate new retention time windows for each standard on each GC column and whenever a new GC column is installed. The data must be retained by the laboratory.

7.4.2 A retention time window is calculated for each analyte and surrogate by using the initial calibration check standard (CCV) of each sequence. The windows shall be +/- 0.10 minutes around the absolute retention time for each analyte and surrogate. Analytes are identified when peaks are observed in the RT window for the compound.

7.5 Sample Quality Control for Preparation and Analysis. The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank (MB), matrix spike (MS), a duplicate (MSD), a laboratory control sample (LCS), and a duplicate (LCSD) in each analytical batch and the addition of surrogates to each field sample and QC sample.

7.5.1 The suggested surrogate is 4-bromofluorobenzene (BFB). Additional or different surrogates may be used to conform to project specific requirements. The surrogate should be spiked at a level consistant with the mid-point of the instrument calibration range.

7.6 Before processing any samples, the analyst should demonstrate, through the analysis of an instrument blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, an instrument blank should be analyzed as a safeguard against chronic laboratory contamination.

7.6.1 All blanks must be free of target agents or the agents must be below the PQL.

7.7 Documenting the effect of the matrix should include the analysis of one matrix spike (MS)/matrix spike duplicate (MSD) pair and one laboratory control sample (LCS)/laboratory control sample duplicate (LCSD) pair.

7.7.1 The following compounds should be used in the MS/MSD and LCS/LCSD QC samples: 1,4-Dithiane, 1,4-Thioxane, and HD. Additional or different compounds may be used to conform to project specific requirements. These compounds should be spiked at a level consistant with the mid-point of the calibration range.

7.7.2 The spiking solutions should not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

7.7.3 Great care must be taken to maintain the integrity of all standard solutions. It is recommended all standards be stored at minus 4C or less, in amber bottles with PTFE-lined screw-caps.

7.7.4 A Laboratory Control Sample (LCS) and duplicate (LCSD) must be included with each analytical batch. The LCS/LCSD consist of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. When the results of the matrix spike analysis indicate a potential problem because of the sample matrix itself, the LCS/LCSD results are used to verify that the laboratory can perform the analysis in a clean matrix. The average and range of the percent recoveries will be maintained and plotted.

7.7.4.1 All recoveries of the LCS and LCSD must be within current QC limits for recovery (%Rec.) and relative percent difference (RPD).

7.7.4.1.1 The RPD is calculated as follows:

$$RPD = \frac{|LCSA - LCSDA|}{\frac{1}{2}(LCSA + LCSDA)} \times 100$$

where:

LCSA = amount detected in the LCS
LCSD = amount detected in the LCSD

7.7.4.2 If any recovery fails to meet its criterion, the failing extract must be analyzed again. If the LCS or LCSD fails yet again, it is deemed out-of-control and the entire batch must be re-extracted and re-analyzed.

7.7.4.3 All averages and RPDs should be within current control limits. Out-of-Statistical-Control is defined as one or more of the following:

- An average that is outside either the upper or lower control limit.
- A RPD value that is above the upper control limit.
- Eight (8) or more successive average recovery points on the same side of the central line.

7.7.4.4 If an out-of-statistical-control situation occurs, the analyst must examine the analytical system for unusual conditions and should perform routine maintenance as needed in an attempt to determine the cause of the situation. Recalibration of the analytical system may be required.

7.8 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. The surrogate recovery must meet the criteria describe in Appendix I.

7.9 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc.

7.10 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

7.11 A summary of all quality control criteria is listed in Appendix II.

8.0 REPORTING REQUIREMENTS AND ORDER OF DATA DELIVERABLES

8.1 The analyst shall provide reports and other deliverables as specified in this section unless superceded in writing by a client-specified format. The required content and form of each deliverable is described in this section. All reports and documentation must be:

- Legible
- Clearly labeled and completed in accordance with instructions in this section
- Arranged in the order specified in this section
- Copies must be legible
- Handwritten corrections of the information must be legible, initialed, and dated.
- White-out and Post-It® notes are not allowed

8.1.1 Prior to submission, the analyst shall arrange items and the components of each item in the order listed in these sections.

8.2 The Sample Data Package is divided into the eight major units described in this section. The Sample Data Package shall include data for the analyses of all samples in one Sample Delivery Group (SDG), including field samples, dilutions, re-analyses, blanks, Laboratory Control Samples/ Laboratory Control Samples Duplicate(s) (LCS/LCSD), and any requested or required Matrix Spike/Matrix Spike Duplicate(s) (MS/MSD). A blank, colored (preferably blue) sheet of paper will separate sections.

8.2.1 Section 1

8.2.1.1 Narrative: This document shall be clearly labeled "ECBC Monitoring Branch Analytical Narrative" and shall contain at a minimum:

- Laboratory name
- Client/Project name
- ECBC sample numbers in the SDG
- SDG number
- Batch number(s)

- Detailed documentation of any quality control, sample, shipment and/or analytical problems encountered in processing the samples reported in the data package.
- Discussion of any IOP modification/variance.

8.2.1.2 Injection log covering the entire analytical sequence. If more than a single log is necessary, forms shall be arranged in chronological order by instrument.

8.2.1.3 Scratch Log complete with analyst, instrument, date extracted, date analyzed, and sample results. After supervisor review, this is moved to Section 8 and the clearance log is placed in this section.

8.2.1.4 QC Charts of average LCS %Rec. and RPD for each target analyte.

8.2.1.5 Surrogate Recovery Form. This form includes all QC, samples, dilution, and re-extractions.

8.2.1.6 Method Blank Summary. This form includes all QC, samples, dilution, and re-extractions associated with a method blank. If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank, by instrument.

8.2.2 Section 2

8.2.2.1 GC/MS tune verification. This form is used to report the results of the GC/MS instrument tune verification. For each ion listed on the form, report the percent relative abundance.

> *NOTE: For BFB, one or more of the high mass ions may exceed the abundance of the ion listed on the form as the nominal base peak, m/z 95 for BFB. Despite this possibility, all ion abundances shall be normalized to the nominal base peak listed.*

If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.

8.2.2.2 Initial calibration data, area report, and plots of linear regression shall be included in chronological order by instrument, if more than one instrument is used. Required items are standard(s) quantitation reports and reconstructed ion chromatograms for the initial (five-point) calibration if the initial calibration was performed in the same analytical sequence. If no initial calibration was performed, then only the area report and plots for the current calibration are required. Spectra are not required, however, EICPs displaying each manual integration are (Section 8.3.1).

8.2.3 Section 3

8.2.3.1 Check Standards data and evaluation report shall be included in chronological order by instrument, if more than one instrument is used. The Check Standard Evaluation form must be followed by the quantitation report and the reconstructed ion chromatograms. Spectra are not required, however, EICPs displaying each manual integration are (Section 8.3.1).

8.2.4 Section 4

8.2.4.1 Matrix Spike/Matrix Spike Duplicate Recovery (MS/MSD) data and evaluation form. If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the MS/MSDs, by instrument. The MS/MSD form must be followed by the quantitation report and the reconstructed ion chromatograms. Spectra are not required, however, EICPs displaying each manual integration are (Section 8.3.1).

8.2.5 Section 5

8.2.5.1 Method, reagent, and instrument blanks in chronological order, by instrument. All false positive detections must be Q-deleted. The quantitation report and the reconstructed ion chromatograms plus spectra for all detected and non-detected agents shall be provided.

8.2.6 Section 6

8.2.6.1 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) data and evaluation form. If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank, by instrument. The LCS/LCSD form must be followed by the quantitation report and the reconstructed ion chromatograms. Spectra are not required, however, EICPs displaying each manual integration are (Section 8.3.1).

8.2.7 Section 7

8.2.7.1 Sample data, including dilutions, and re-analyses data, shall be arranged in packets with the quantitation report and the reconstructed ion chromatograms plus spectra for all detected and non-detected agents. The samples shall be placed in increasing Monitoring Branch sample number order, considering both letters and numbers. This may not correspond to the order in which the samples were analyzed.

8.2.8 Section 8

8.2.8.1 Chain of Custodies, copy of standard logbook pages of all standards used in the analyses, and copy of extraction log book showing all samples in the SDG.

8.2.8.2 Any other supporting data. For example, screening data, memos, notations.

8.3 Data and Report Formats

8.3.1 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS system must identify such edits or manual procedures. A hardcopy printout of the Extracted Ion Current Profile (EICP) of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all target agents and surrogates.

8.3.2 The analyst shall identify all samples, including dilutions and re-analyses, Laboratory Control Sample(s)/ Laboratory Control Sample Duplicate(s), Matrix Spike/Matrix Spike Duplicate(s) (MS/MSD), blanks, and standards with a unique Monitoring Branch sample number.

8.3.2.1 For field samples, the Monitoring Branch sample number is the XXX digit unique identifying number assigned at log-in to the MUD tracking system. In order to facilitate data assessment, the analyst shall use the following sample suffixes:

- XXXXX = Monitoring Branch sample number
- XXXXXMS = Matrix spike sample
- XXXXXMSD = Matrix spike duplicate sample
- XXXXXRE = Re-extracted and re-analyzed sample
- XXXXXDL = Sample analyzed at a dilution
- XXXXXDL2 = Sample analyzed at a secondary dilution
- XXXXXDL3 = Sample analyzed at a third dilution
- XXXXXDUP = Sample duplicate

8.3.2.2 The Monitoring Branch sample number shall be unique for each blank and LCS/LCSD within an SDG. The unique number will be the eight digit extraction batch number (as described in SOP#XXX) plus the following suffixes:

- XXXXXXXX = extraction batch number
- XXXXXXXXMB = Method blank
- XXXXXXXXLCS = Laboratory control sample
- XXXXXXXXLCSD = Laboratory control sample duplicate

8.3.3 Cross out unused columns and spaces. Initial and date all cross outs.

8.3.4 Do not use paper clips or staple pages together.

8.3.5 A summary of the data format and order can be found in Appendix III.

TABLE 1
CHARACTERISTIC MASSES (m/z) FOR CHEMICAL WARFARE AGENTS

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
GA	106	117, 133
GB	125	99, 81
GD (1 & 2)	126	99, 82
GF	99	67, 54
HD	158	160, 109
HN-1	120	122, 92
HN-3	154	156, 63
L	212	151, 86
VX	114	127, 167
1,4-Thioxane	104	61, 74
1,4-Dithiane	120	127, 167
CEES	124	75, 126
TDG	104	61, 45
TDP	150	61, 93

TABLE 2
BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA

m/z	Required Intensity (relative abundance)
50m/z	8 to 40% of m/z 95
75m/z	30 to 66% of m/z 95
95m/z	Base peak, 100% relative abundance
174m/z	Greater than 50% to 120% of m/z 95
176m/z	Greater than 93% but less than 101% of m/z 174

Appendix I

It is necessary that the laboratory evaluate surrogate recovery data from individual samples. The analyst must evaluate the recovery to determine if acceptable system performance was maintained for each analysis. Surrogate limits are established by the laboratory annually, or as specified by clients or programs. The surrogate recovery is calculated as:

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

If the recovery is not within surrogate recovery limits, the following procedures are necessary:

- Check to be sure that there are no errors in the calculations or surrogate solution standard. If errors are found, recalculate the data accordingly.
- Rerun the extract (to ensure a proper injection). If it fails again, the sample must be re-extracted (in a new batch with new QC) and re-analyzed.
- If the re-extraction passes, then it is assumed there was a spiking error and the initial analysis is not reported. If it fails yet again, then it is assumed there is a matrix affect and both (the initial and re-extraction) analyses are reported.

Appendix II

Typical analytical sequence

With initial calibration:
1. tune with 1 - 50ng BFB
2. instrument blank
3. 5-point calibration
4. calibration verification
5. method blank
6. LCS/LCSD
7. samples, MS/MSD
8. CCV (every 10 samples)
9. instrument blank (optional)
10. ending calibration check standard

Without initial calibration:
1. tune with 1 - 50ng BFB
2. instrument blank
3. calibration verification
4. method blank
5. LCS/LCSD
6. samples, MS/MSD
7. CCV (every 10 samples)
8. instrument blank (optional)
9. ending calibration check standard

QC Criteria

- Tune: GC/MS system must meet all criteria before any analysis (Table 2).
- Initial Calibration: linear regression (do not force zero). Correlation coefficient (r^2) ≥ 0.990 (section 6.4.5.2.1.1).
- Calibration verification/check: +/- 20% (25% for VX and L) difference (section 6.5.2).
    - If calibration check is less that 20% (25% for VX and L), all samples prior to the calibration check must be reanalyzed. If the calibration check is greater than 20% (25% for VX and L), analysis must stop however previous samples maybe reported.
- Surrogate: all surrogate recoveries must be within current lab limits. If not, sample must be re-analyzed. If it fails again, the sample must be re-extracted and re-analyzed (Appendix I).
- LCS/LCSD: %Rec. and RPD must be within current lab limits. If not, re-analyze. If either one fails again, the entire batch must be re-extracted. Sample analysis can not begin until the LCS/LCSD meet all criteria (section 7.7.4).
- MS/MSD: recoveries are evaluated against current laboratory limits, however, if any recovery is outside these limits there is no correction action. All failures will be discussed in the report narrative.

Appendix III
Data Package Format

- Section 1
  - Narrative
  - Checklist
  - Scratch log
  - Injection log
  - QC Charts
  - Surrogate summary
  - Method Blank summary

- Section 2 (Calibration)
  - Tune verification data in chronological order
  - Initial Calibration data in chronological order, by instrument
    - Area report and plots
    - Raw data(on day that a calibration is analyzed – no spectra except for manual integrations)

- Section 3 (Check standards)
  - Check standards in chronological order
    - Check standard evaluation report
    - Quant report/chromatogram(no spectra except for manual integrations)

- Section 4 (MS/MSD)
  - MS/MSDs in chronological order, by instrument
    - MS/MSD evaluation report
    - Quant report/chromatogram(no spectra except for manual integrations)

- Section 5 (Blanks)
  - Blanks in chronological order
  - Detailed report for each blank

- Section 6 (LCS/LCSD)
  - LCS/LCSDs in chronological order, by instrument
    - LCS/LCSD evaluation report
    - Quant report/chromatogram(no spectra except for manual integrations)

- Section 7 (Analyses – in Monitoring Branch sample # order)
- Quant report/ Chromatogram (with label)
    - Final quant report (after Qdeleting false hits)
    - Manual spectra/blowups of target agents

- Section 8 (support data)
  - COCs
  - Logbook pages (extraction/standards)
  - Any other information (i.e. memos, notes, support data)